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AUSTENITIC STAINLESS ALLOYS

Their Properties and Characteristics—Part I

By Vsevolod N. Krivobok and Rush A. Lincoln

Abstract

Eighteen thirty-pound ingots were made with carbon content of approximately 0.05 and 0.15 per cent and with chromium ranging from 17-19 per cent and nickel from 7-9 per cent for each series of carbon content.

These ingots were reduced by hot and cold rolling to strip 0.038 inch thick, which was annealed to dissolve any carbides. The effect of composition on the mechanical properties was determined on both annealed strip and strip after different degrees of cold rolling.

The ultimate strength and elongation in both cold-rolled and annealed samples change sharply with composition and appear to be influenced by the stability of the austenite with respect to ferrite formation during the cold work imposed by the tensile test.

Correlation of the ultimate strength with the hardness of different alloys showed a variation in the annealed condition, that became less pronounced with increasing amounts of cold work.

Bend tests on the annealed and on cold-rolled samples indicate the superiority of the high carbon alloys for use in forming operations after cold rolling to high tensile strengths.

The proof stress of these alloys, in the annealed condition, is low but upon cold rolling, increases more rapidly than the ultimate strength. With 30 per cent cold reduction a proof stress greater than half the ultimate strength resulted in all of the alloys studied.

IT has become almost a rule that the application of an alloy of promise, in various engineering projects, precedes a comprehensive study of the basic properties of such an alloy; lamentable circumstance, indeed often resulting in incorrect deductions, thus retarding, if not

A paper presented before the Eighteenth Annual Convention of the American Society for Metals, Cleveland, October 19 to 23, 1936. Vsevolod N. Krivobok is Professor of Metallurgy, Carnegie Institute of Technology, Pittsburgh, and Associate Director of Research, Allegheny Steel Co., Brackenridge, Pa. Rush A. Lincoln is a member of the Research Laboratories, Allegheny Steel Co., Brackenridge, Pa. Manuscript received June 25, 1936.

altogether arresting, the progress of new applications and attainments. Though this statement should not be taken literally, it will be conceded that the implications are correct. Design, fabricating problems and considerations of safety justify the constructor in setting up certain specifications. The metallurgist should definitely know whether the properties of the material—it would be better to say "properties

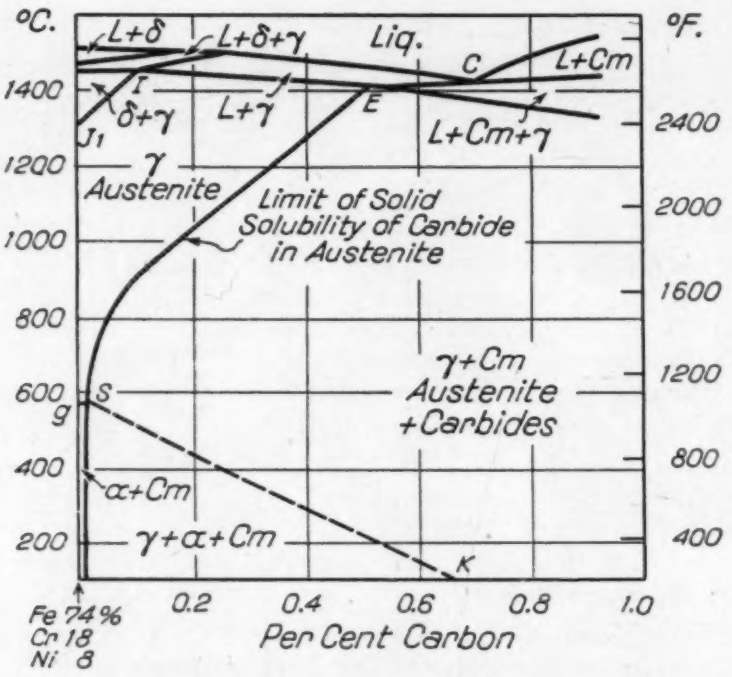

Fig. 1—Influence of Carbon on Steels Containing 18 Per Cent Chromium and 8 Per Cent Nickel (from The Book of Stainless Steels).

and characteristics"—will meet, with relative ease, the specifications. Two words, "relative ease" will explain, we believe, our statement concerning the implications. They will further absolve us from the accusation of having discussed the obvious. Austenitic stainless alloys have not escaped the general rule referred to above.

An understanding of the nature of the alloys should make us suspect that their properties must be influenced by the concentrations of alloying elements. It is known that these alloys are readily rendered austenitic by simple, rapid, cooling from high temperatures; thus, after this treatment we find them to consist of a single phase. In such circumstances the presence of only one phase does not necessarily denote true equilibrium conditions. First indication of this was given when the well-known phenomenon of carbide precipitation was discovered. Continued work further disclosed the probability—

now definitely established—that alloys of the general 18-8 type should consist, if at equilibrium at room temperature, of austenite (Cr-Ni-Fe-C solid solution), ferrite (also a solid solution) and complex carbides. Neither the distribution of alloying elements between these various phases, nor the relative amount of phases are established. Even the diagram itself, reproduced in Fig. 1, is strictly tentative, showing, as it were, the relative positions of various fields and co-existent phases, but claiming no accuracy in the positions of temperature and concentration boundaries.

Even if equilibrium conditions in an alloy system called for a single phase over a range of concentrations it is to be expected that the properties of such phase may vary, in some cases quite considerably. This is true in those ferrous alloys in which, through the addition of definite amounts of alloying element, an alpha iron allotropic form is the equilibrium phase at all temperatures. Familiar examples are alloys of iron and chromium, iron-silicon, etc.[1]

Less is known about the alloys in which the high temperature phase (gamma iron) is retained at room temperature. We do know definitely[2] that this phase is not an equilibrium one and that either by means of cold working, prolonged heating at proper temperatures or by the combination of the two, it can be readily decomposed. We also know that the degree of decomposition, under similar conditions of treatment, is governed by the composition. With increasing concentration of certain alloying elements, the alloys are rendered more and more stable—if the reader will allow this expression.

The dependence of mechanical properties upon concentration (we speak of "austenitic" alloys only) has not been carefully ascertained. Perhaps the reason is that the "instability" of the alloys under certain treatment has not been realized.

The austenitic type of chromium-nickel-iron alloys found a large field of application in the form of sheet and strip in various gages, hot-rolled, cold-rolled or hot-rolled and annealed.[3] If subjected to the last treatment, the material possesses all of the familiar characteristics of austenite; namely, typical, well-known structure,

[1]V. N. Krivobok, "Alloys of Iron and Silicon," Alloys of Iron Research Monograph Series. "Alloys of Iron and Chromium," TRANSACTIONS, American Society for Metals, Vol. 23, March 1935, p. 1.

[2]As, for example, in high manganese steels. See as a reference, "A Study of the Constitution of High Manganese Steels," V. N. Krivobok, TRANSACTIONS, American Society for Metals, Vol. XV, No. 6, p. 893, also series of articles "Alloys of Iron and Manganese," published in TRANSACTIONS, American Society for Metals between 1930 and September 1935.

[3]"Annealed" is a shop expression: in reality it means fully softened by heating to 1850-2050 degrees Fahr. and cooled in air.

para-magnetism, absence of critical transformation, and consequently, nonsusceptibility to heat treatment. However, changes in properties will be noted if an alloy is subjected to cold deformation: for example, various degrees of cold working, which, in alloys of certain composition may be quite slight, render the alloys of the 18-8 type[4] definitely ferromagnetic. The influence of nickel is known to be that of a "stabilizer" for the austenitic character of the alloys. Increasing nickel content in the alloys of 18 per cent chromium and usual amounts of carbon results in the reduced tendency for decomposition as is well illustrated in Table I, in which the phenomenon of decomposition is detected through the determination of magnetic permeability.

Two alloys, one with 18 per cent chromium, 8 per cent nickel and the other containing 18 per cent chromium and 15 per cent nickel, were subjected to cold rolling to a certain per cent reduction and immediately tested for permeability, with results shown in Table I.[5]

Table I

Original Gage	Final Gage	Per Cent Reduction	Permeability at 200-H	
			Alloy 18-8	Alloy 18-15
0.083	0.083	0	1.023	Less than 1.01
0.083	0.066	20	1.797	—
0.083	0.050	40	3.495	—
0.083	0.033	60	5.350	Less than 1.01
0.083	0.017	80	8.570	—

The composition of alloy 18-8 was 18.47 per cent chromium, 8.20 per cent nickel and 0.11 per cent carbon. Alloy 18-15 contained practically the same chromium: namely, 18.61 per cent chromium, 0.07 per cent carbon and 15.62 per cent nickel. Disregarding slight variation in carbon, it becomes evident that an alloy with 15.6 per cent nickel is infinitely more stable, at least insofar as the formation of magnetic ferrite from nonmagnetic austenite by means of cold working is concerned. Hence, in the range of compositions, let us say from 8 to 15 per cent nickel, maintaining the other elements constant, we obtain a number of alloys of various "degrees of stability"—we already apologized for the use of this superfluous expression.

Since it must be admitted that the result of decomposition is the formation of a different phase—it is to be expected that the properties

[4]"18-8" is a name adopted in America for alloys of Cr-Ni class, containing, generally, 17-20 per cent Cr., 7-10 per cent Ni., and up to 0.20 per cent C.

[5]The authors are indebted to V. W. Van Epps of the Research Staff of Allegheny Steel Co., for this test.

of alloys suceptible, in various degrees, to decomposition will be different. In fact the expectancy seemed logical that with proper adjustment in the ratio of alloying elements, certain combination of physical properties—let us say strength and ductility—could be obtained, the same combination being unobtainable in alloys of other compositions. And since the solution of any engineering problem is dependent upon the above-mentioned combination of properties, scientific curiosity was augmented by the possibility of supplying the engineer and the designer with definite and dependable data, on the strength of which the stainless materials could be used in those installations where now their use is questioned and erroneously avoided.

Curiously enough—considering the importance of chromium-nickel stainless alloys—the available literature contains little information concerning their physical properties. The most extensive investigation on cold rolling of 18-8 type alloys and the interdependence of several variables, including composition, degree of cold work, degree of hardenability prior to heat treatment, etc. was published by Pfeil and Jones.[6]

Bain and Aborn, Pilling, Monypenny, Sergeson, Schmidt and Jungwirth, Forstman, among others, have investigated cold working of chromium-nickel alloys of various compositions. No definite attempt is shown to have been made to isolate and analyze the influence of each element not only on the hardenability, but also on the resulting mechanical and other properties. When such attempts were made, merely as a matter of interest, the selected compositions varied too widely in more than one element. Thus, it is not exaggerating to sum up our knowledge of today by quotation, perfectly justifiable when it was made in 1929: "By severe cold working of 18-8 a very marked increase in tensile strength and hardness is obtained, but with a continual decrease in ductility." It is, however, too general and too incomplete.

Experimental Procedure

When alloys vary rather widely in composition, as for example, 18 per cent chromium, 8 per cent nickel, and 18 per cent chromium, 15 per cent nickel—alloys selected for magnetic tests cited above—it is reasonable to expect that the influence of slight difference in composition of other elements present (or the fact that samples

[6] "A Contribution to the Study of the Properties of Austenitic Steels," *Journal*, British Iron and Steel Institute, 1933, Vol. 77, No. 1, p. 337.

were taken from two commercial heats, separately made) would be negligible.

It seemed advisable, however, to avoid this variation (however slight) in the projected investigation of progressive increase in chromium, nickel and carbon content. Consequently, all of the alloys described below were made from two commercial heats in ingots weighing about thirty pounds, the increase in percentages of the above-mentioned elements being accomplished by addition of necessary ferro-alloys. Thus, manganese, silicon, phosphorus, sulphur content, as well as that of oxygen and nitrogen were kept constant. The variable of melting practice was also eliminated.

The carbon content, designated as "high" and "low" was selected as 0.13-0.14 and 0.05-0.06 per cent. Chromium and nickel varied in steps of 1 per cent, sufficient number of alloys being made to cover all possible combinations. Table II shows the intended and actual analysis of each alloy.

The material listed in Table II was processed as follows: Ingot forged to sheet bar, 1 inch thick, ground, hot-rolled to 0.080 inch. Low carbon material was annealed at 1950 degrees Fahr. (1056 degrees Cent.) for 10 minutes, air cooled, while the high carbon alloys were annealed at 2050 degrees Fahr. (1120 degrees Cent.) for 20 minutes and quenched in water. After the usual pickling, all sheets were cold-rolled to 0.038 inch thick and again annealed at 1950 or 2050 degrees Fahr. (1065-1120 degrees Cent.) according to carbon. From there on the alloys were cold-rolled to the desired thickness and tested.

Table II
Analysis of Alloys Tested

Type	Per Cent Intended	Carbon Actual	Per Cent Intended	Nickel Actual	Per Cent Intended	Chromium Actual
17-7 L.C.	0.06	0.053	7	7.07	17.00	17.13
17-8 L.C.	0.06	0.051	8	8.05	17.00	16.94
17-9 L.C.	0.06	0.050	9	9.79	17.00	16.83
18-7 L.C.	0.06	0.048	7	7.23	18.00	17.99
18-8 L.C.	0.06	0.055	8	8.51	18.00	17.62
18-9 L.C.	0.06	0.052	9	9.80	18.00	17.66
19-7 L.C.	0.06	0.056	7	7.33	19.00	18.68
19-8 L.C.	0.06	0.050	8	8.17	19.00	18.63
19-9 L.C.	0.06	0.051	9	9.14	19.00	18.22
17-7 H.C.	0.13	0.140	7	7.07	17.00	17.26
17-8 H.C.	0.13	0.140	8	8.22	17.00	17.00
17-9 H.C.	0.13	0.130	9	9.65	17.00	17.08
18-7 H.C.	0.13	0.140	7	6.96	18.00	17.98
18-8 H.C.	0.13	0.140	8	7.70	18.00	17.80
18-9 H.C.	0.13	0.130	9	8.56	18.00	17.64
19-7 H.C.	0.13	0.160	7	6.68	19.00	19.68
19-8 H.C.	0.13	0.160	8	7.90	19.00	18.68
19-9 H.C.	0.13	0.160	9	8.78	19.00	18.88

A word in regard to selected annealing procedure. The tensile strength of cold-rolled (or even hot-rolled), annealed chromium-nickel steels is influenced by the temperature of anneal and the time at temperature; alloys reach maximum "softening" at a certain temperature and change their tensile strength very little, if any, with subsequent increase of 200-300 degrees Fahr. in annealing temperature. Higher temperature will lower the tensile strength, presumably because of grain growth. The results below illustrate the point.

Annealing Temperature Degrees Fahr.	Tensile Strength Pounds per square inch
1700	107,400
1800	106,200
1850	93,200
1900	93,600
1950	93,300
2000	92,700
2050	91,400
2100	90,800

As a further check on the proper anneal, a count of grain size was made and found uniform in all alloys, ranging from No. 6 to No. 8.[7]

The selection of proper annealing temperature has to be carefully considered. In the case of high carbon alloys it is imperative to ascertain that the metal is free from grain boundary carbides; this was checked by microscopic examination and modified Krupp Test. Occasional checks were also made on the low carbon series.

There is another important point. When alloys are in their optimum soft condition, at least one variable—and there are many—in the subsequent judgment of the characteristics of the alloys is eliminated. Further, it is obvious that only under such condition can the method of producing material of any given properties be always duplicated.

While the influence of nickel (from the work of Pfeil and Jones) is understood, at least qualitatively, the role played by chromium and carbon has not been definitely established.

In the future discussion pertaining to this subject, we shall frequently use the term "stability" or refer to alloys as being "stable" or "unstable." These terms express the different degrees of susceptibility of alloys toward further change in their constitution when subjected to identical treatment. The above-mentioned susceptibility

[7]Grains per square inch at 100 diameters $= 2^{n-1}$, where "n" is the index number.

should be governed by concentrations of chromium, nickel and carbon as we learned from the studies on the constitution of these and related alloys. The purpose of this investigation was to determine how these changes in constitution are reflected in the properties and characteristics of the alloys. Also, to study those ranges of composition where, because of obvious instability, the influence of a single element was most pronounced.

To execute this problem, we have deliberately selected alloys with widely different carbon content; namely, 0.06 or less, and 0.14-0.16 per cent carbon. First, we shall discuss alloys of low carbon in which nickel and chromium vary in steps of one per cent. Then we shall take up high carbon alloys.

Results

A. *Low Carbon Alloys*—In Fig. 2 are reproduced the results of cold rolling to predetermined gages (for each composition) of a series of constant chromium (17 per cent) and carbon (0.05 per cent), but variable nickel (7, 8 and 9) alloys.* It will be observed at once that nickel has a most profound effect on the properties of this particular type of alloys even in the annealed condition. A variation in nickel content of about 2¾ per cent (from 7 to 9¾ inches) decreases the tensile strength from 132,000 to 85,000. It should be remembered that the alloys are in the softest condition available after heat treatment. The elongation is also most markedly affected, being 22 and 62 per cent, respectively. This abnormal variation will soon be explained and the influence of other factors pointed out.

Upon cold working, the increase in strength is quite regular. In fact, the relative difference in strength in the annealed condition of alloys 17-8 and 17-9 is maintained throughout the cold working operation; reduction of 60 per cent by cold rolling will result in a tensile strength increase from 85,000 to 177,000 and from 107,000 to 208,000 pounds per square inch. On the other hand, although we started with widely different elongations, we arrived, after the mentioned 60 per cent cold reduction, at the same figure for both alloys—a figure so low that for some fabricating purposes, the material is not suitable. Thus, this class of alloys, if required for some good reason, should not be selected for tensile strength above a certain value. If moderately high strength is required, different degrees of ductility are obtainable

*In these tensile tests a head speed of $\frac{5}{32}$ of an inch per minute was used throughout.

by the proper selection of nickel content in alloys with carbon of 0.06 per cent or less. For example; if 150,000 pounds per square inch is desired, an alloy can be furnished with 12, 17, or 20 per cent elongation. The corresponding bending and forming characteristics of the alloys, also differing, will be discussed later.

Fig. 3 depicts the results secured on alloys of the next series

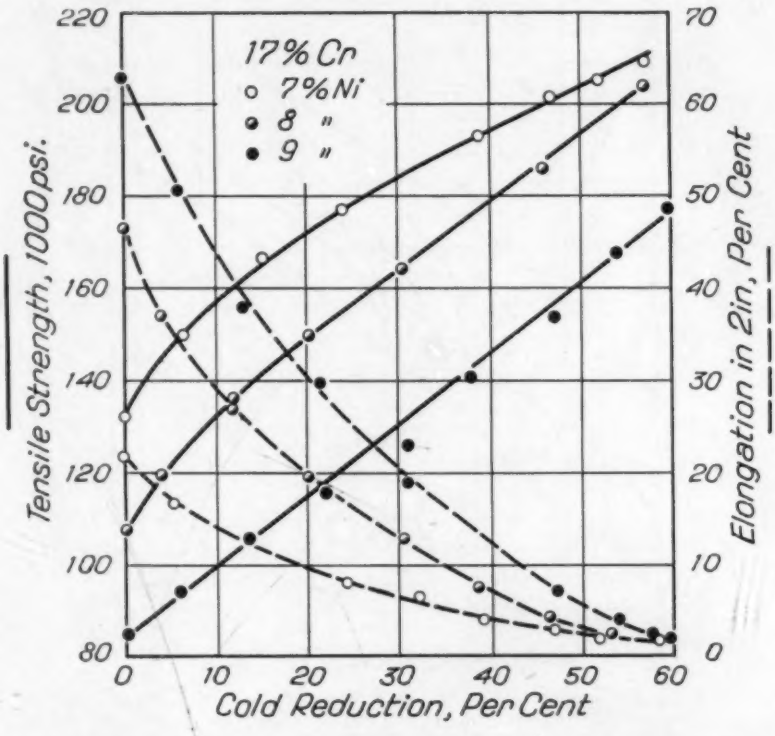

Fig. 2—Summary of the Tensile Strength and the Elongation of Alloys Containing About 0.05 Per Cent Carbon, 17 Per Cent Chromium, and from 7 to 9 Per Cent Nickel, After Various Amounts of Cold Work.

with 18 per cent chromium, 0.05 per cent carbon and nickel again varying from 7 to 10 per cent. The tensile strength of annealed sheets is governed, quite definitely, by nickel content, as is shown by the actual values: 85,000 pounds per square inch for the alloy with 10 per cent nickel and 125,000 pounds per square inch for an alloy with only 7.3 per cent nickel. The proportional regular increase in tensile strength upon cold rolling is maintained for all three alloys. The curves for elongation do not follow the same relationship as previously described (curve No. 2), but they do show the same final results; namely, that elongation figures for all three alloys become similar and very low.

In these series of alloys, the elongations available at 150,000 tensile strength would be 12, 19, and 26 per cent.

The third series, alloys with 19 per cent chromium, 0.05 per cent carbon, and nickel 7.3, 8.2, and 9.1 are shown with their properties in Fig. 4. As was observed in previous cases, the tensile strength "as annealed" and for a given chromium and carbon (less than 0.06

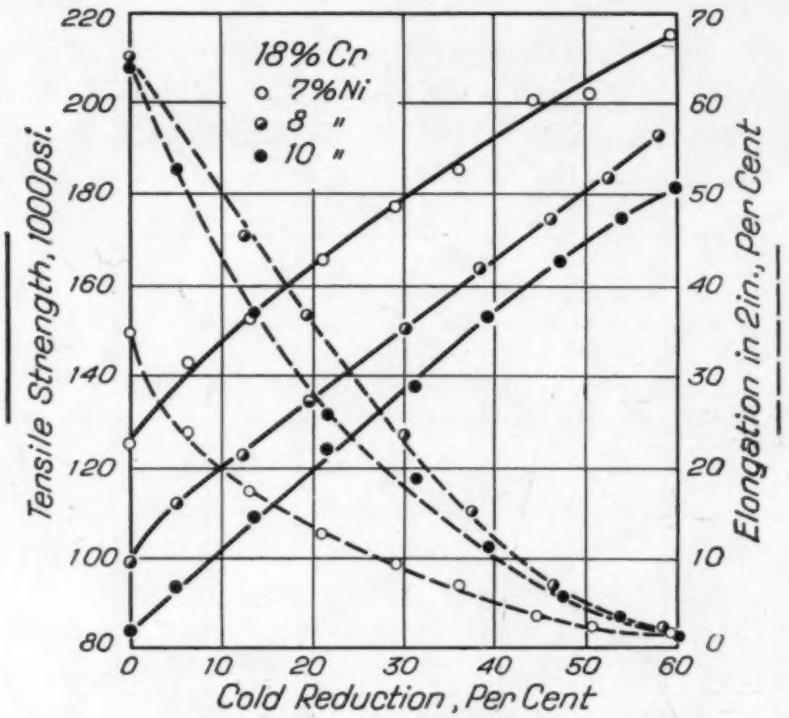

Fig. 3—Summary of the Tensile Strength and the Elongation of Alloys Containing about 0.05 Per Cent Carbon, 18 Per Cent Chromium, and 7 to 10 Per Cent Nickel, After Various Amounts of Cold Work.

per cent) is a function of nickel content. The elongation is not proportional to tensile strength: alloys 19-8 and 19-9 exhibit the same elongation although their tensile strength is different by some 12,000 to 13,000 pounds. While in the case of each alloy, cold working produces proportional increase in tensile strength, elongation values again come down to very low values, previously observed. In this series, for a selected tensile strength of 150,000 pounds per square inch we may have elongations of 23, 20, and 7 per cent.

This analysis of the results tells us that in alloys of constant carbon content, namely, 0.05 per cent, and of the constant tensile strength of 150,000 pounds per square inch we may obtain elongation values ranging from 7 per cent minimum to 26 per cent maximum by adjusting, within narrow limits of 3 per cent, the concentrations of

chromium and nickel. It is to be shown that the absolute values for elongation do not constitute the whole story. But it will be deferred until later.

A clear picture of the characteristics of the alloys can be had from Fig. 5 in which are plotted the relationship between tensile strength (or elongation) and chromium content of those alloys in

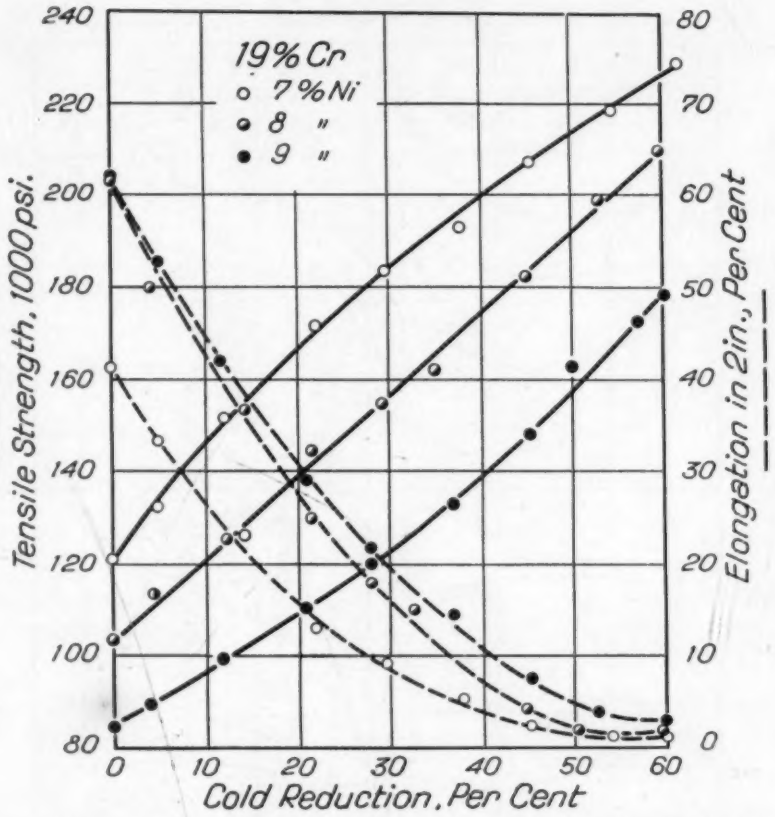

Fig. 4—Summary of the Tensile Strength and Elongation of Alloys Containing About 0.05 Per Cent Carbon, 19 Per Cent Chromium, and 7 to 9 Per Cent Nickel, After Various Amounts of Cold Work.

which nickel and carbon are kept constant. Figs. 5, 6, and 7 give this relationship for alloys with 7, 8 and 9 per cent nickel and 0.05 per cent carbon.

The following deductions are borne out by the above figures: In alloys with 7 per cent nickel, the increase in chromium content does not affect the tensile strength in the annealed state, but results in much higher ductility as judged by elongation. With the increased amount of cold deformation the tensile strength increases more rapidly in alloys with higher chromium content. The elongation, although remaining higher for a certain amount of cold reduction, falls

off more rapidly upon further work until all three alloys become equally devoid of ductility. These alloys, incidentally, are of the "border" type, of high initial strength and of a certain degree of "instability." Microscopical examination does not reveal typical austenitic structure even in the properly heat treated samples. Markings not unlike what we are accustomed to call "martensite" are plainly

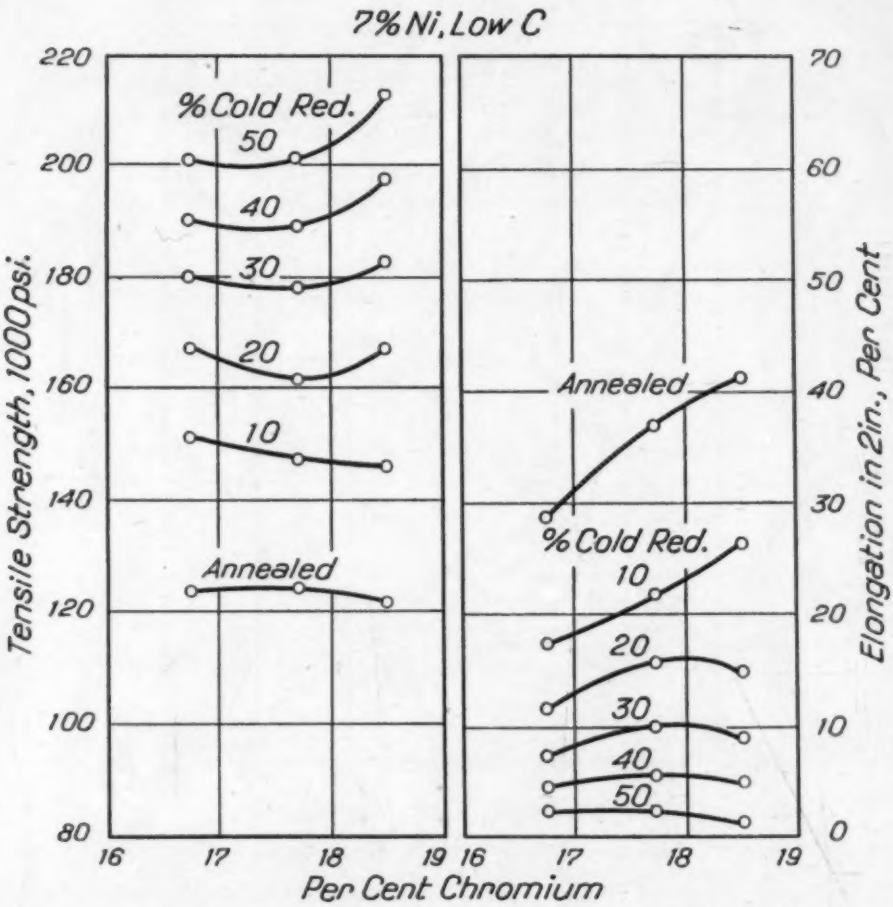

Fig. 5—Influence of Chromium, in Alloys Containing About 0.05 Per Cent Carbon and 7 Per Cent Nickel, Upon the Tensile Strength and Elongation, After Various Amounts of Cold Work.

visible in Fig. 7a. Therein, we believe, lies the explanation of high tensile strength. Although these alloys are readily decomposed by cold distortion, it is not too speculative to assume that the process of their decomposition may not be the same as that of other alloys of different composition and different initial degree of stability.

With the increase in nickel to 8 per cent (Fig. 6) the tensile strength (in the annealed condition) is slightly falling off with the increase in chromium, but the elongation increases markedly. Upon

cold working to a certain reduction, relative values for tensile strength are retained. This is shown even more conclusively in alloys with 9 per cent nickel (Fig. 7). It is to be noted (Fig. 7) that an alloy with 18.5 per cent chromium and 9.0 per cent nickel has the tensile strength which we usually attribute to austenitic chromium-nickel alloys.

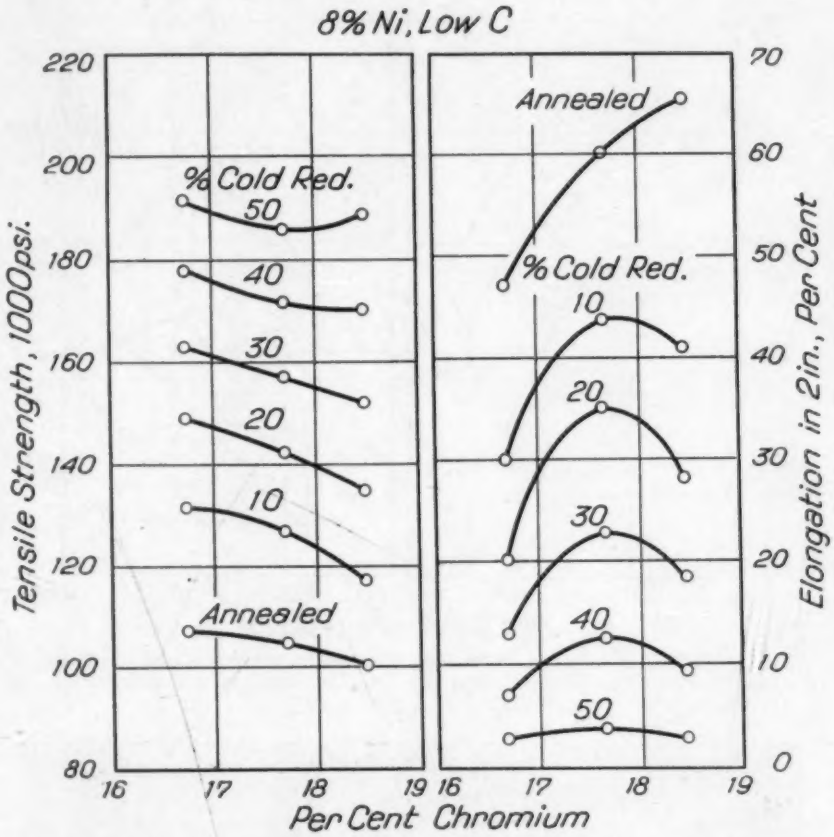

Fig. 6—Influence of Chromium, in Alloys Containing About 0.05 Per Cent Carbon and 8 Per Cent Nickel, Upon the Tensile Strength and Elongation, After Various Amounts of Cold Work.

As to the elongation for both 8.0 per cent and 9.0 per cent nickel alloys, the influence of chromium as an alloying element is not regular; with increasing chromium ductility goes through a maximum up to certain degree of cold work.

In general, the effect of chromium and nickel on the stability of austenite is a complex phenomenon. The addition of chromium to iron, or for that matter, iron-nickel, or even iron-nickel-carbon alloys promotes the formation of ferrite as a stable phase. The influence of additions of nickel is reverse: in iron, iron-chromium, or iron-chromium-carbon alloys it promotes the formation of austenite,

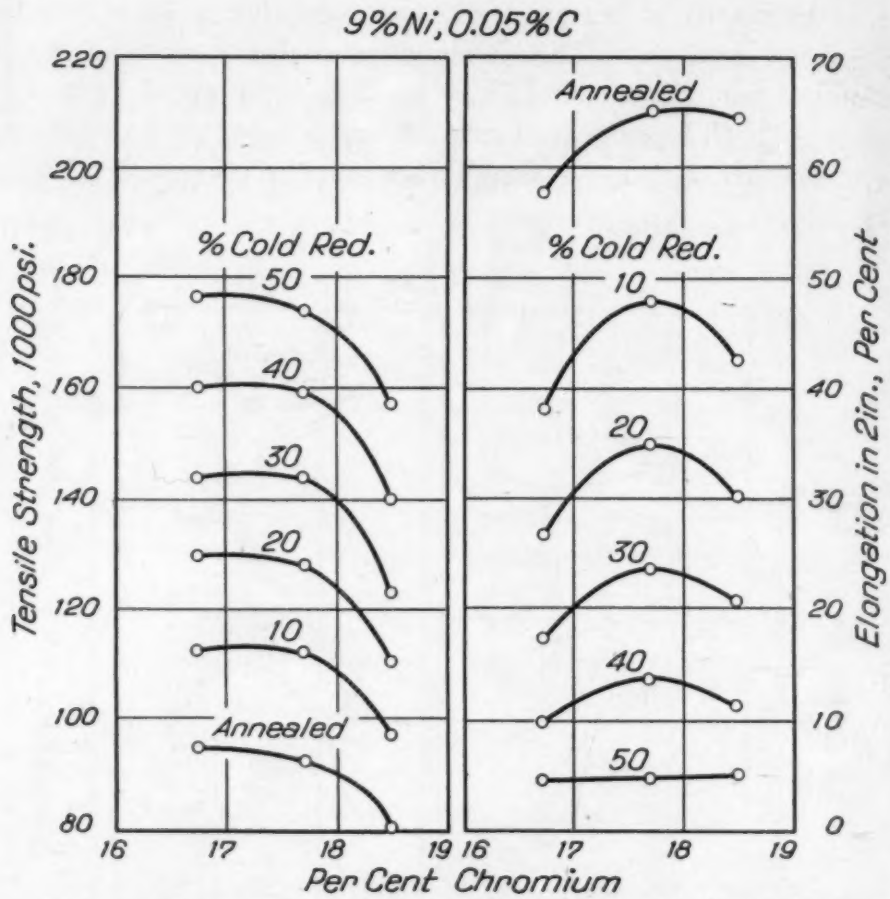

Fig. 7—Influence of Chromium, in Alloys Containing About 0.05 Per Cent Carbon and 9 Per Cent Nickel, Upon the Tensile Strength and Elongation, After Various Amounts of Cold Work.

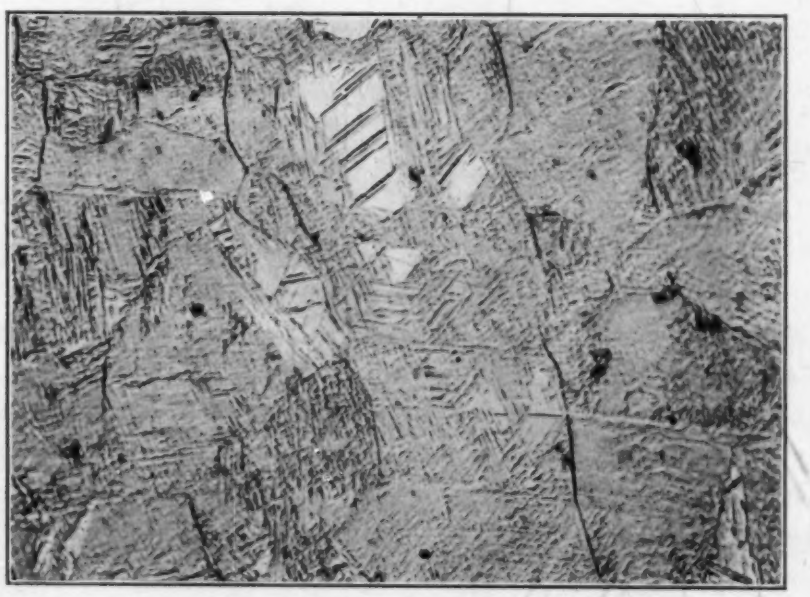

Fig. 7a—Showing the Widmanstätten Markings, in an Alloy Containing 17 Per Cent Chromium, 7 Per Cent Nickel and 0.05 Per Cent Carbon, After Being Heated at 1950 Degrees Fahr. and Air Cooled. × 500.

extends its stability range to lower temperatures and to wider ranges in composition. But both chromium as well as nickel have a profound effect on the rate of formation of the stable (equilibrium) phase; the greater their concentration the slower the rate. These opposing influences should be remembered when we attempt to evaluate the "instability" of alloys after cold deformation. The degree of

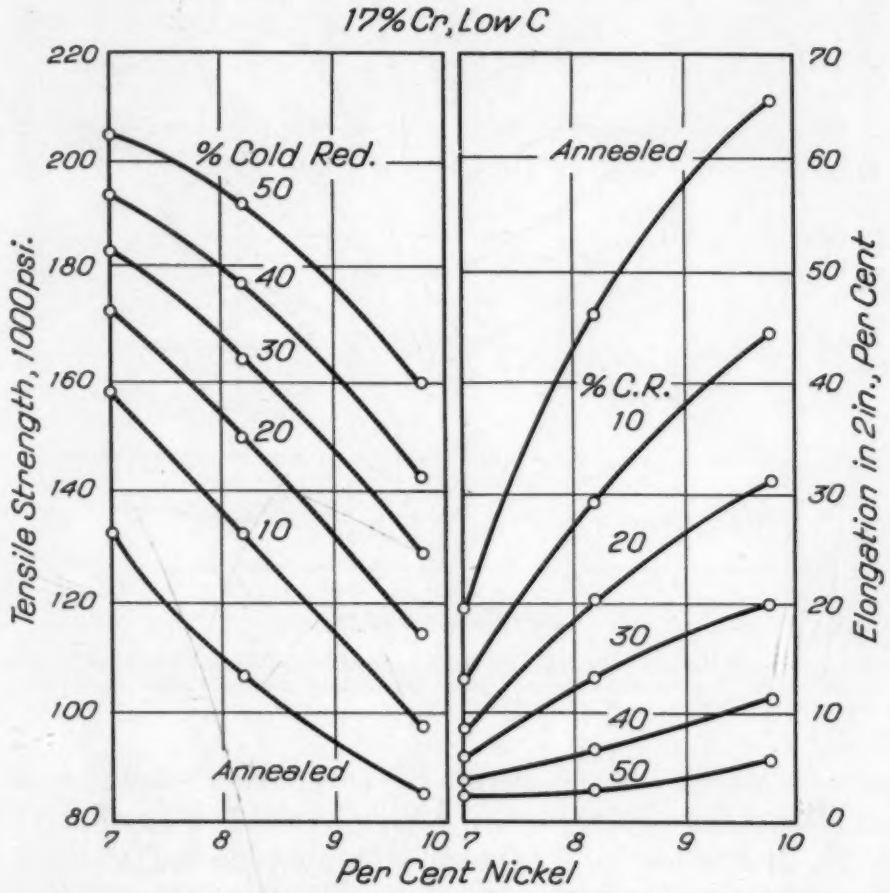

Fig. 8—Influence of Nickel, in Alloys Containing About 0.05 Per Cent Carbon and 17 Per Cent Chromium, Upon the Tensile Strength and Elongation, After Various Amounts of Cold Work.

"instability" may differ, depending not so much on the actual amount of any one element, as the total amount of elements present. The influence of carbon is most pronounced as will be shown later. Furthermore, the initial constituents of the alloy, difficult to identify in case of a "border alloy" as described and illustrated by the photomicrograph Fig. 7a in the previous discussion, may also regulate the actual stages of decomposition. Thus the observations based on experimental facts should not appear anomolous.

Returning to our discussion of alloys with constant chromium

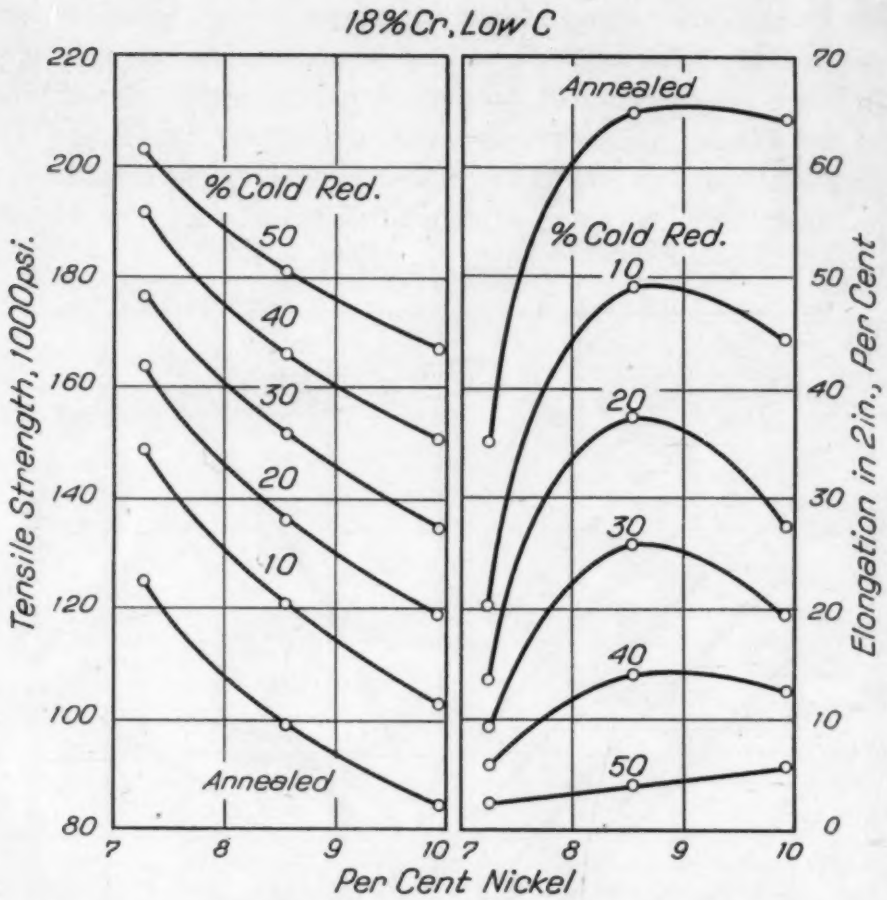

Fig. 9—Influence of Nickel, in Alloys Containing About 0.05 Per Cent Carbon and 18 Per Cent Chromium, Upon the Tensile Strength and Elongation, After Various Amounts of Cold Work.

and carbon (less than 0.06 per cent) the effect of nickel can be shown by means of similar curves which are reproduced in Figs. 8, 9, and 10. With an increase in nickel content, within the limits stated, the tensile strength of fully softened alloys is very markedly lowered and these relative values for strength are maintained throughout the cold working process. Careful examination of the results discloses a rather interesting difference in the response of the alloys toward cold deformation. Alloys of the lowest nickel concentration (7 per cent), regardless of the chromium content, increase in tensile strength by smaller and smaller increments as the material is cold reduced by the same amount. In alloys of highest nickel content (9 per cent) the increments of increase in tensile strength are quite regular. This can best be illustrated by the experimental results collected in Table III. The latter alloys (i.e., those with 9 per cent nickel) also show quite remarkable consistency in the actual values for tensile strength

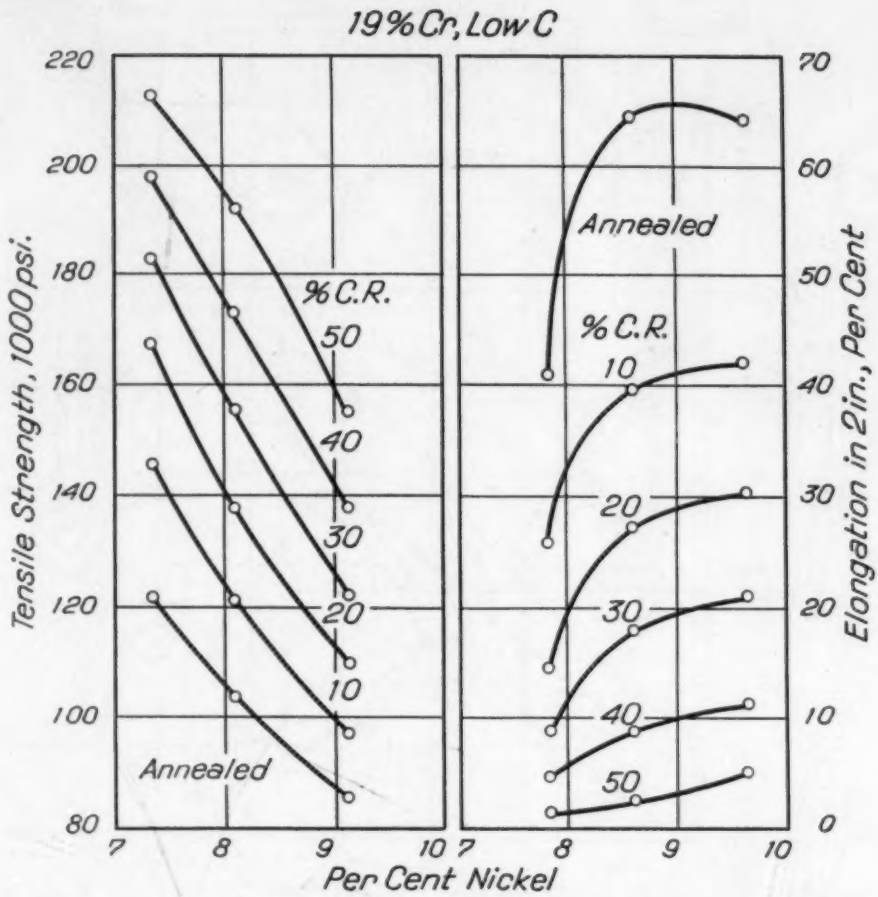

Fig. 10—Influence of Nickel, in Alloys Containing About 0.05 Per Cent Carbon and 19 Per Cent Chromium, After Various Amounts of Cold Work.

not only in the material "as fully softened," but also at any stage of reduction by cold deformation. Attentive examination of the curves will show that it is true regardless of chromium content: the alloys may be said to be approaching the same degree of "stability." This, of course, has already been made evident from previous curves (Figs. 5, 6 and 7), but here we point out actual rather than relative values.

The trend in the measured precentages of elongation is shown in the same Figs. 8, 9 and 10. Since the data for these three figures

Table III

Increase in Tensile Strength—Pounds Per Square Inch—Chromium-Nickel Alloys

	17–7	18–7	19–7	17–9	18–9	19–9
First ten per cent cold reduction	25,000	21,000	24,000	14,000	18,000	12,000
Second ten per cent cold reduction	15,000	16,000	22,000	17,000	16,000	13,000
Third ten per cent cold reduction	11,000	12,500	16,000	14,000	16,000	12.000
Fourth ten per cent cold reduction	10,000	16,000	14,000	14,000	16,000	15,000
Fifth ten per cent cold reduction	10,000	11,000	15,000	17,000	16,000	17,000

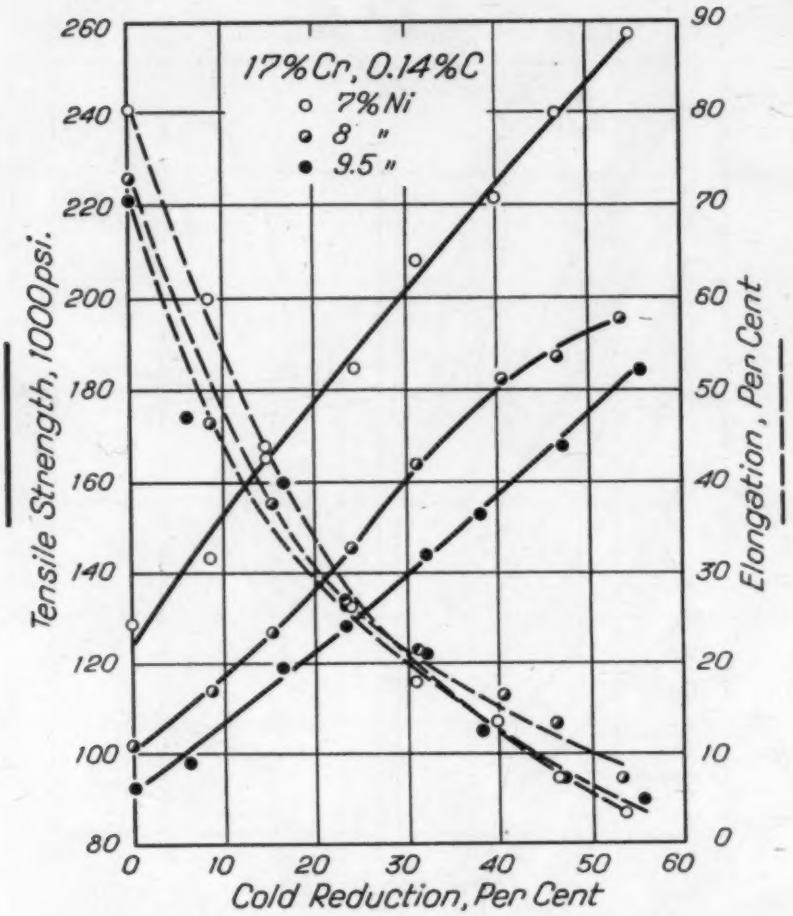

Fig. 11—Summary of the Tensile Strength and the Elongation of Alloys Containing About 0.14 Per Cent Carbon, 17 Per Cent Chromium and 7 to 9.5 Per Cent Nickel, After Various Amounts of Cold Work.

were replotted from the previous Figs. 5, 6 and 7, the probable explanation need not be repeated again.

B. *High Carbon Alloys*—The results of testing high carbon alloys are shown in Figs. 11, 12 and 13. As in previously described work (on low carbon alloys) the results are grouped according to chromium content; namely, 17, 18 and 19 per cent.

For the first series,—those with 17 per cent chromium—the reader will consult Fig. 11. An increase in nickel content has a marked influence on the tensile strength of the alloys "as annealed,"—the higher the nickel the lower the tensile strength, but the values for elongation differ in much less pronounced manner. In fact, allowing for inevitable, small inaccuracies (in measuring elongation after rupture has taken place) the elongation figures for the alloys of 17-8 and 17-9.5 should be considered as practically identical. Alloy 17-7

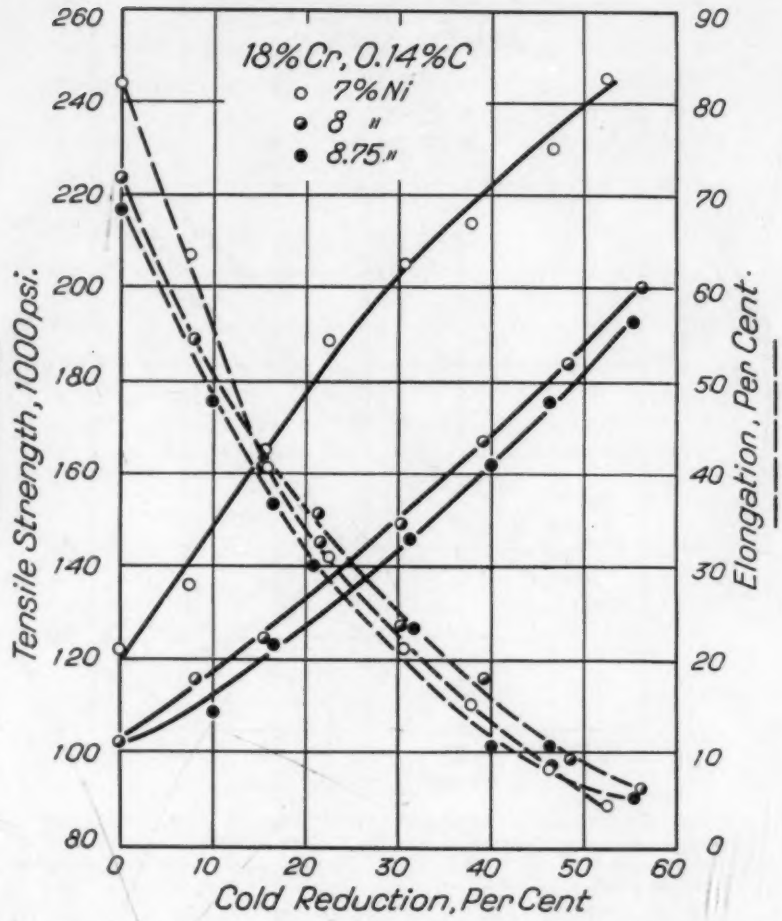

Fig. 12—Summary of the Tensile Strength and Elongation of Alloys, Containing About 0.14 Per Cent Carbon, 18 Per Cent Chromium, and 7 to 8.75 Per Cent Nickel, After Various Amounts of Cold Work.

has been found to possess higher ductility with higher tensile strength.

The latter observation holds true for the alloys with 18 per cent chromium (Fig. 12); in both cases just discussed, an alloy with 7 per cent nickel stiffens (hardens, strengthens) upon cold working at a somewhat greater rate than alloys with either 8 or 9 per cent nickel. (This matter of hardenability will be discussed in detail later). When chromium is increased to 19 per cent (in one or two alloys slightly higher) and carbon to 0.16 per cent—the reader is referred to Fig. 13—the values for tensile strength (in the annealed state) are so similar that, apparently, the effect of nickel is almost nullified. This last observation, checked by numerous tests, is at variance with stated views regarding the "stabilizing of austenite" influence of nickel in low carbon alloys. Thus, again, we arrive at the conclusion

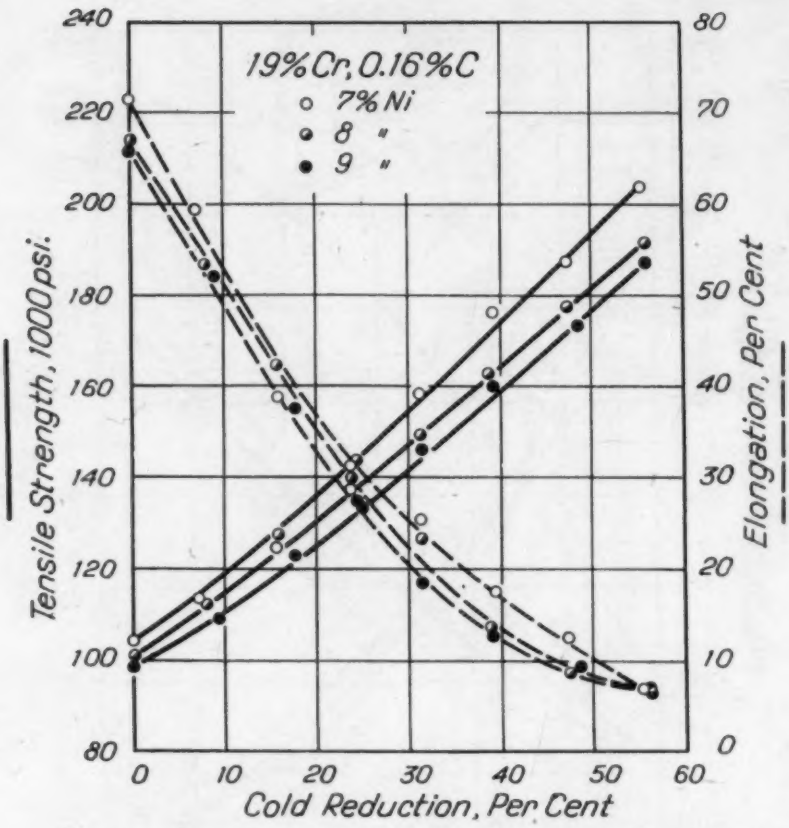

Fig. 13—Summary of the Tensile Strength and Elongation of Alloys, Containing About 0.16 Per Cent Carbon, 19 Per Cent Chromium, and 7 to 9 Per Cent Nickel, After Various Amounts of Cold Work.

that it is not one single element that determines the mechanical properties of chromium-nickel alloys but the sum total of the elements chromium, nickel and carbon and their relation to each other. The possible—and probable—reasons have already been discussed and will not be repeated here.

It would be of interest to follow the procedure, already adopted, and tabulate the ductility, measured by elongation, in alloys of equal tensile strength, but of different analysis. If we choose, quite arbitrarily, the strength of 200,000 pounds per square inch the following values for elongation, in alloys containing 0.14-0.16 per cent carbon, will be available:

Alloy type	17-7	17-8	18-7	18-8	19-7
Elongation in 2 inches—Per Cent	22.5	8.0	25.0	6.0	8.0

Or for the tensile strength of 175,000 pounds per square inch.

Elong. in 2 inches —Per Cent	17-7	17-8	17-9.5	18-7	18-8	18-9	19-7	19-8	19-9
	35.0	17.0	7.0	37.0	13.0	8.0	16.0	11.0	7.0

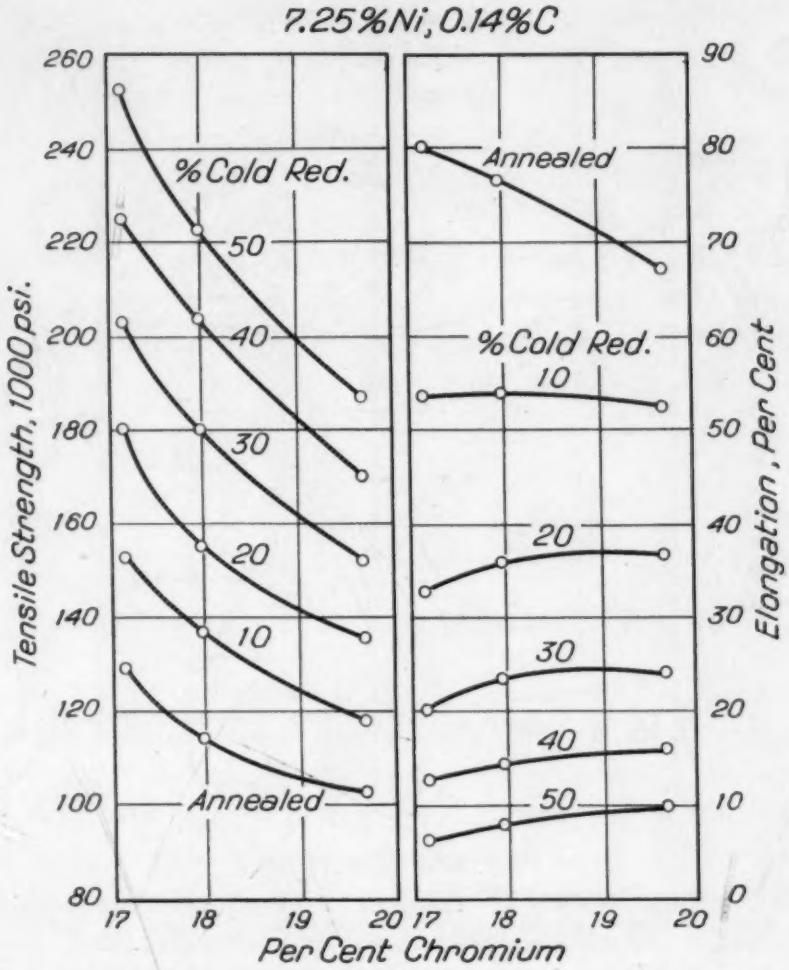

Fig. 14—Influence of Chromium, in Alloys Containing About 0.14 Per Cent Carbon and 7.25 Per Cent Nickel, Upon the Tensile Strength and Elongation, After Various Amounts of Cold Work.

A rather astonishingly wide range of attainable percentages of elongation! Other characteristics, as for example, bending, forming, etc. should bear certain relationship to the cited mechanical properties. But let us not deviate from orderly discussion: this particular question will be taken up later.

It is with the purpose of facilitating to the interested reader the judgment of the presented data that we include graphical pictures of the influence of chromium (Figs. 14, 15 and 16) and of nickel (Figs. 17, 18 and 19).

Fig. 14 (together with Figs. 5 and 6) bring out the vast influence of chromium on alloys of high carbon as contrasted with the results obtained on low carbon alloys. It will be remembered that in low carbon, 7 and 8 per cent nickel series, the tensile strength of

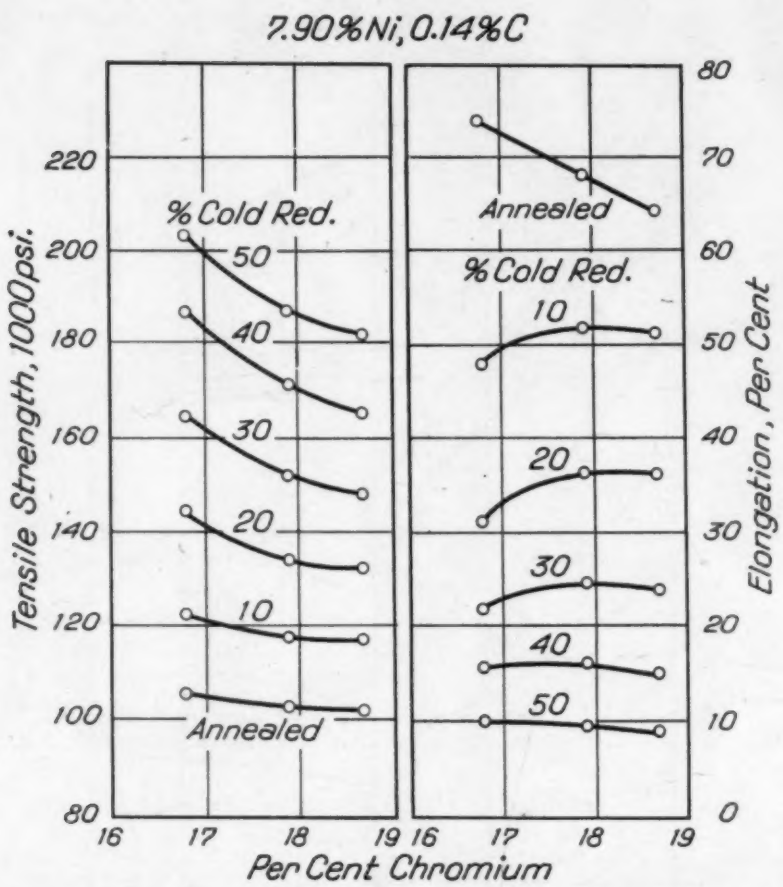

Fig. 15—Influence of Chromium, in Alloys Containing About 0.14 Per Cent Carbon and 7.9 Per Cent Nickel, Upon the Tensile Strength and Elongation After Various Amounts of Cold Work.

fully softened alloys was practically independent of the concentration of chromium: that is to say, a wide latitude in chromium analysis could be permitted without altering tensile properties. In high carbon alloys, variations in chromium content result in widely different mechanical characteristics, which, furthermore, appear to be intensified by the cold rolling process. This is not surprising. The solid solubility for carbon is exceeded in the alloys under investigation and the deformation of the material is accompanied by the breaking down of pseudo solid solution. The process of breaking down is indeed a complex one involving changes in the nature of the matrix and perhaps influenced by the nature of the deformation. It is rather surprising, that the difference in properties should be maintained—and even intensified as we mentioned—throughout the rolling process.

With higher nickel content (Fig. 15, nickel 8 per cent and Fig. 16, nickel 8.60-9 per cent) we find that the influence of chromium rapidly diminishes. As in the low carbon series, the alloys approach,

as has been suggested, a more stable form and therefore attain the properties characteristic of such form.

Fig. 17, 18 and 19 was replotted, as was suggested, for the convenience of the reader, to isolate and depict graphically the influence of nickel. To judge progressive influence of carbon, we must have additional data (now in progress of accumulation) secured from alloys of medium carbon, that is, about 0.10 per cent. However, it is evident from presented studies that, as a general rule, alloys of a similar tensile strength will exhibit better ductility with increased carbon content and will stand more cold processing before reaching optimum values.

PART II

By V. N. Krivobok, R. A. Lincoln and R. Patterson, Jr.

A statement was made at the beginning of this discussion that not much printed information is available concerning the tensile properties of the "austenitic" chromium-nickel alloys. We had, however, a very pleasant opportunity to discuss this matter with many friends, both metallurgists and design engineers. Often the opinion was expressed that the values for tensile strength are fictitious, because the metal hardens while it is tested, that is, cold deformed by the tensile machine. The same reasoning, of course, applies to testing any other alloy, the difference being only that of degree. But this is beside the point. It is readily granted that the statement has certain justifications. Hence, we were told it is of utmost importance to determine the elastic properties, the familiar terms "proportional limit," "yield point" are deliberately avoided—in these alloys. In the fully annealed condition, the alloys are quite notorious for their low elastic properties, while in the cold-worked state the determination of elastic behavior is most difficult. Mehl and Gensamer, who diligently studied this question, express their belief, shared by the authors, that "in cold-worked materials," to quote them,[8] "the elastic

[8] R. F. Mehl and M. Gensamer, "X-Ray Studies of Luders' Lines and Strain Figures in Low Carbon Steels," *Metals and Alloys*, June, 1935.

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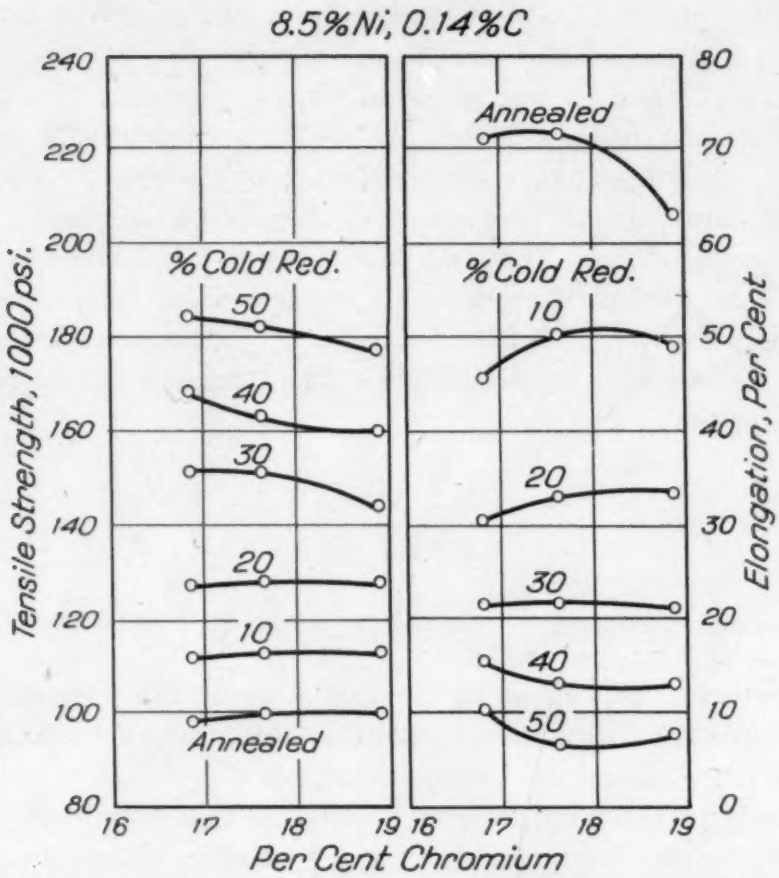

Fig. 16—Influence of Chromium, in Alloys Containing About 0.14 Per Cent Carbon and 8.5 Per Cent Nickel, Upon the Tensile Strength and Elongation, After Various Amounts of Cold Work.

range is very restricted, the yield point is ill-defined, and in no portion of the stress-strain curve may an elongation at constant load be found." This circumstance necessitated the acceptance of an arbitrary method of testing, such as adopted by the A.S.T.M. for material "whose stress-strain diagram in the region of yield is a smooth curve of gradual curvature," the method known as "set method,"[9] or the method incorporated in many specifications, among them those of the United States Navy, and referred to as "proof stress." The definition of the "proof stress" is as follows—load in pounds per square inch, which, when released results in a permanent set of no more than 0.0002 inch in 2 inches.[10]

By means of optical arrangement, entirely independent of testing machine and therefore free from common difficulties encountered

[9]See A.S.T.M. Engineering Materials Standards, Sept. 1934, p. 26.

[10]The figure allowed for permanent set may vary: 0.0002 inch are specified by the U. S. Navy.

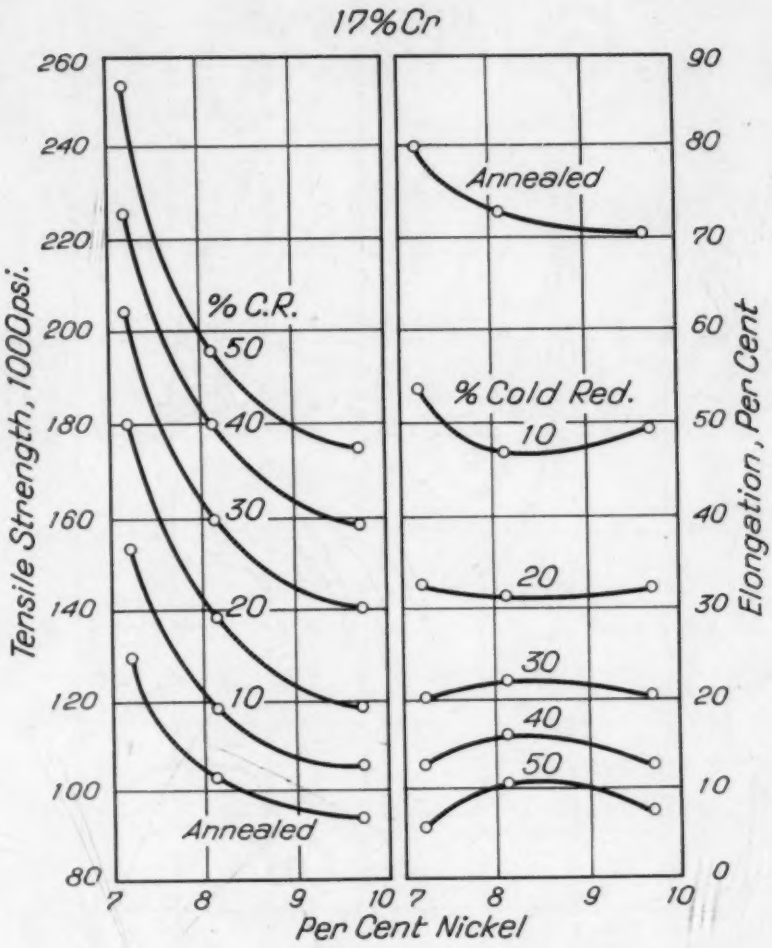

Fig. 17—Influence of Nickel, in Alloys Containing About 0.15 Per Cent Carbon and 17 Per Cent Chromium, Upon the Tensile Strength and Elongation, After Various Amounts of Cold Work.

in the most refined extensometers, the proof stress, in accordance with the above given definition, was determined; the alloys selected were of varying analysis and subjected to different degrees of cold deformation. The readings of the optical arrangement (identical with the one which is used in creep testing) were accurate to 0.00005 of an inch.[11] Fig. 20 shows the results obtained on the alloy with 0.11 per cent carbon, 18.27 per cent chromium, and 7.57 per cent nickel. Each load, as indicated in the figure, was applied to a new sample of the same material, having the tensile strength of 193,000 pounds per square inch. It is interesting to note, in passing, that if the above-mentioned "set-method" is applied to the reproduced curves, identical results are obtained. It should, of course, be ad-

[11]Because of the human element involved, we do not wish to guarantee accuracy better than 0.0001.

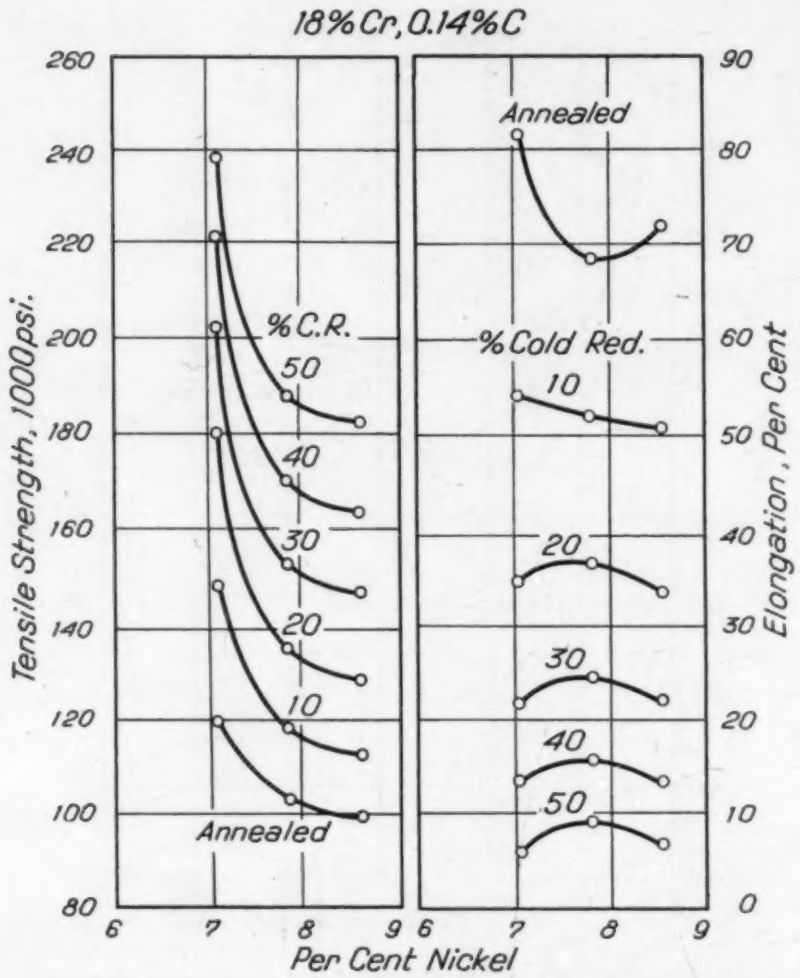

Fig. 18—Influence of Nickel, in Alloys Containing About 0.14 Per Cent Carbon and 18 Per Cent Chromium, Upon the Tensile Strength and Elongation, After Various Amounts of Cold Work.

mitted that the adoption of the proof stress method as a criterion for judging elastic properties of metals may be criticized on scientific grounds. We used it for want of anything better and because of the necessity of supplying engineers with the data they say they need. And since we recognize the stated limitations it would be wise to refrain from the theoretical, and of necessity, speculative discussion as to its basic meaning and dependability. We stress, however, that the figures for proof values, given below, were obtained by the best available method, carefully conducted and repeatedly checked. Certain rather general deductions will therefore be in order.

The values for proof stress (as defined above) will be found in Table IV, in which an attempt was made to arrange alloys according to their composition. Critical study of this table will show, first,

Table IV
Values for Proof Stress

Type Alloy	C.	Cr.	Ni.	Condition	Gage	Per Cent Cold Reduction	Optical Telescope	Automatic Recorder	Tensile Strength in Lbs./Sq. In.	Per Cent Elongation in 2 Inches
16–6 H. C.	0.13	16.47	6.60	C. R.	...	30.0	89,000	.....	213,000	24.0
16–6 H. C.						12.0	50,000	.....	189,000	32.0
17–7 L. C.	0.053	17.13	7.07	Annealed	.037	None	21,000	.....	137,000	21.0
				C. R.	.031	15.0	52,000	.....	161,000	12.5
				C. R.	.025	30.0	110,000	.....	180,000	6.5
				C. R.	.0202	45.0	150,000	.....	197,000	2.0
17–9 L. C.	0.05	16.83	9.79	Annealed	...	None	.....	25,000	87,000	68.0
				C. R.	.0327	15.0	37,000	37,500	107,000	42.0
				C. R.	.0267	30.0	68,000	68,000	126,000	23.0
				C. R.	.021	45.0	86,000	.....	152,000	4.0
19–7 L. C.	0.056	18.68	7.33	Annealed	.0385	None	.....	28,000	127,000	36.5
				C. R.	.0325	15.0	55,000	55,000	154,000	21.5
				C. R.	.0266	30.0	105,000	105,000	177,000	12.0
				C. R.	.021	45.0	148,000	144,000	199,000	2.5
19–9 L. C.	0.051	18.22	9.14	Annealed	.038	None	.....	25,000	86,000	68.5
				C. R.	.0328	15.0	42,500	.....	102,000	44.0
				C. R.	.0263	30.0	60,000	.....	128,000	22.0
				C. R.	.0205	45.0	100,000	.....	155,000	5.5
18–7 Med. C.	0.11	18.27	7.57	C. R.	...	30.0	101,000	.....	193,000	20.0
17–7 Med. C.	0.10	17.05	7.01	C. R.	...	27.0	80,000	.....	192,000	25.0
19–9 Med. C.	0.10	19.11	9.22	C. R.	...	...	85,000	.....	202,000	2.0
17–7 H. C.	0.14	17.26	7.07	Annealed	.0355	None	.....	30,000	136,000	65.0
				C. R.	.030	15.0	62,000	.....	176,000	32.0
				C. R.	.025	30.0	120,000	120,000	206,000	15.5
				C. R.	.020	45.0	160,000	.....	238,000	3.5
17–9 H. C.	0.13	17.08	9.66	Annealed	.0372	None	.....	32,000	90,000	60.0
				C. R.	.0315	15.0	51,000	51,000	112,000	42.0
				C. R.	.026	30.0	65,000	.....	137,000	18.0
				C. R.	.020	45.0	92,000	97,000	158,000	7.5
19–7 H. C.	0.16	19.68	6.68	Annealed	.037	None	.....	29,000	114,000	80.0
				C. R.	.030	15.0	50,000	.....	144,000	50.0
				C. R.	.0247	30.0	78,000	79,000	172,000	32.0
				C. R.	.0197	45.0	123,000	124,000	200,000	17.0
19–9 H. C.	0.16	18.88	8.78	Annealed	.0385	None	.....	30,000	99,600	70.5
				C. R.	.031	15.0	50,000	.....	120,000	38.0
				C. R.	.026	30.0	75,000	.....	150,000	20.0
				C. R.	.0208	45.0	100,000	.....	173,000	7.0

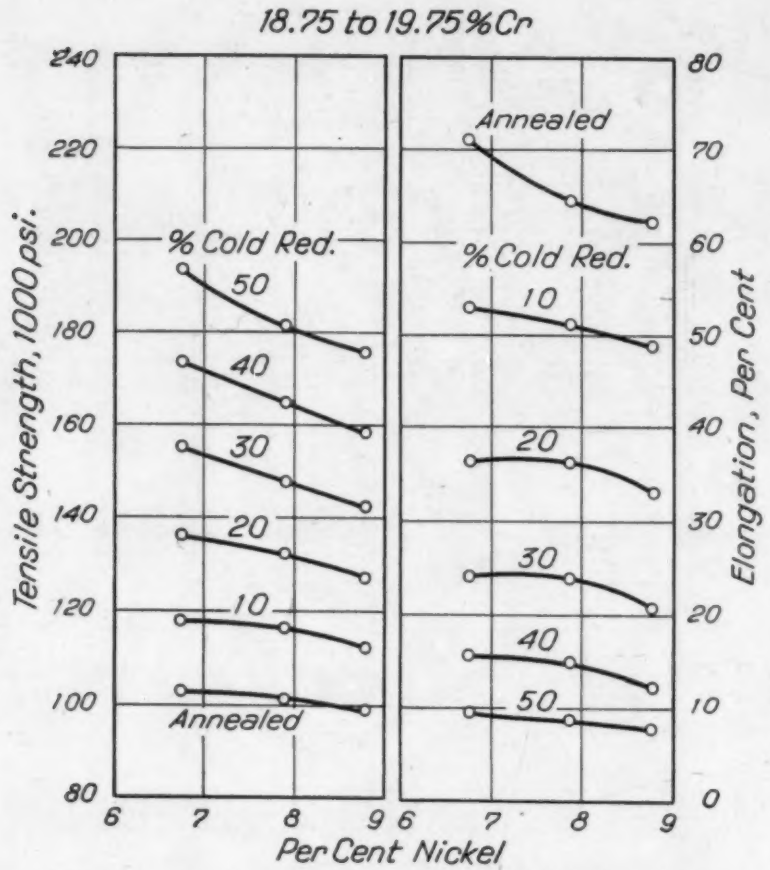

Fig. 19—Influence of Nickel, in Alloys Containing About 0.14 Per Cent Carbon and 18.75 to 19.75 Per Cent Chromium, Upon the Tensile Strength and Elongation, After Various Amounts of Cold Work.

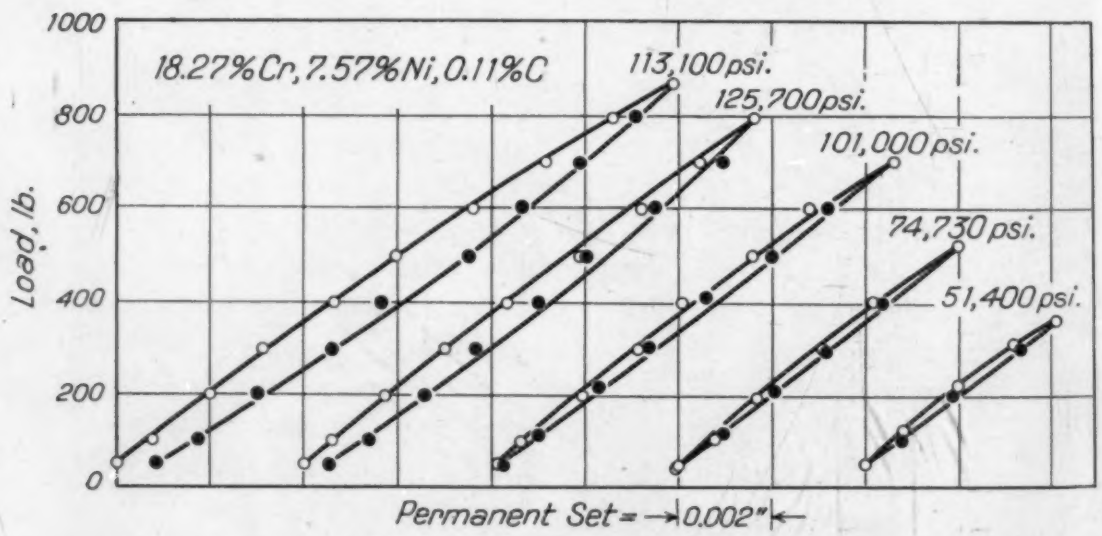

Fig. 20—Stress-Strain Curves Obtained, by Optical Methods, During the Determination of the Proof Stress of an Alloy Containing 18.27 Per Cent Chromium, 7.57 Per Cent Nickel, and 0.11 Per Cent Carbon.

that proof stress values for alloys of a given carbon content and in fully softened condition are, for all practical purposes, independent of the rest of the composition.

Secondly, that the rate of increase of proof stress, induced by cold working, is definitely slower in the more stable than in the less stable alloys, as might have been anticipated.

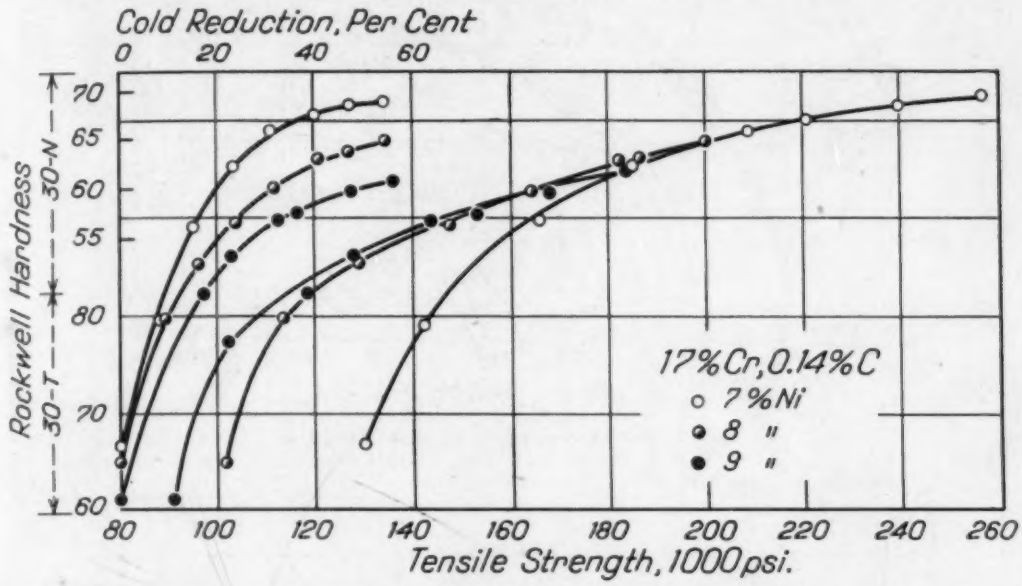

Fig. 21—Summary of the Rockwell Hardnesses of Alloys Containing About 0.14 Per Cent Carbon, 17 Per Cent Chromium, and 7 to 9 Per Cent Nickel, After Various Amounts of Cold Work, and Compared with the Corresponding Tensile Strengths.

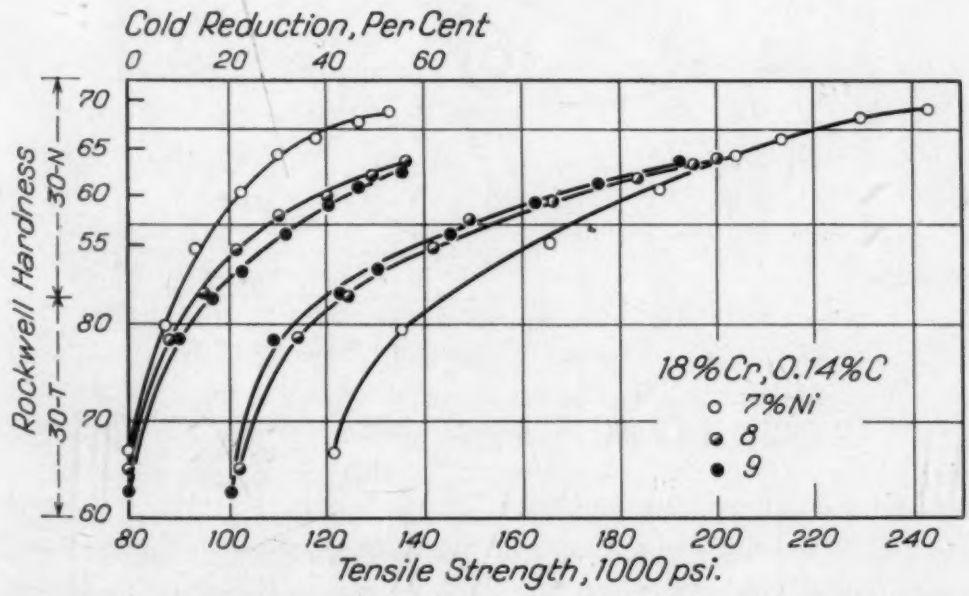

Fig. 22—Summary Similar to that shown in Fig. 21, but for Alloys Containing About 0.14 Per Cent Carbon, 18 Per Cent Chromium, and 7 to 9 Per Cent Nickel.

Finally, another important observation should be pointed out, namely that proof stress is influenced, for a given tensile strength, by the amount of cold work received by the alloy. For example, let us

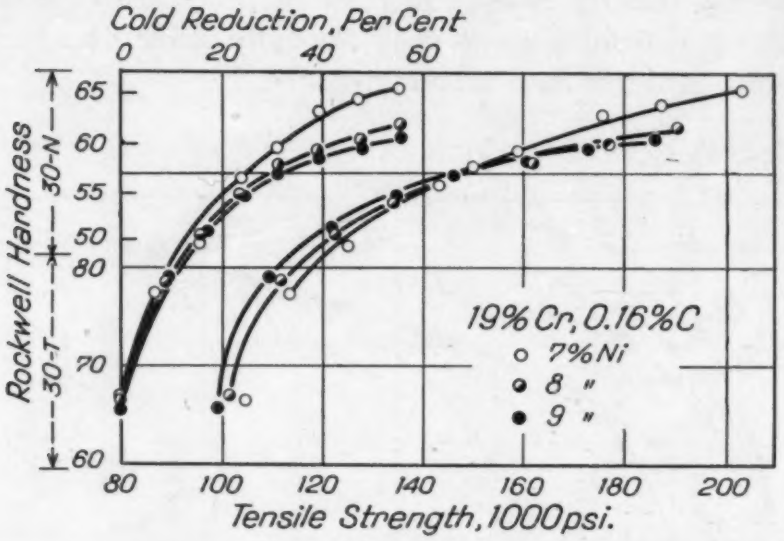

Fig. 23—Same Type of Summary as that Shown in Fig. 21, but for Alloys Containing About 0.16 Per Cent Carbon, 19 Per Cent Chromium, and 7 to 9 Per Cent Nickel.

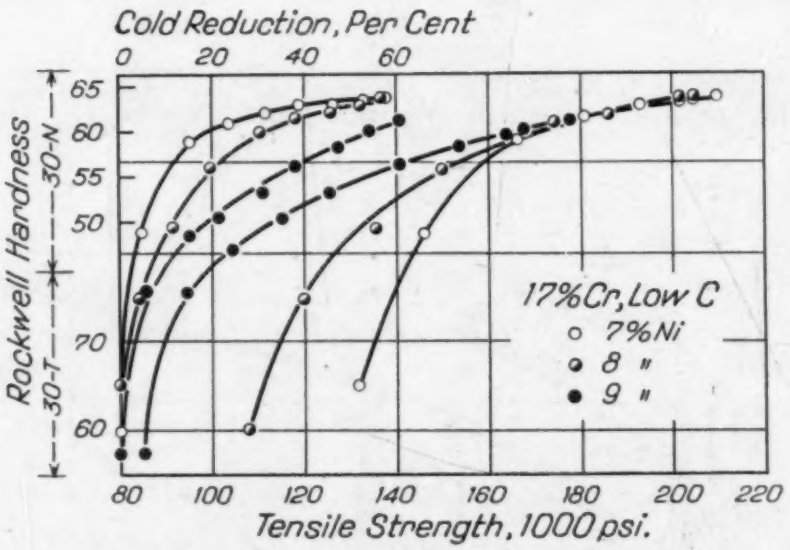

Fig. 24—Same Type of Summary as that Shown in Fig. 21, but for Alloys Containing About 0.05 Per Cent Carbon, 17 Per Cent Chromium, and 7 to 9 Per Cent Nickel.

consider two alloys, low carbon 17-9 and low carbon 17-7. To obtain 152,000 pounds per square inch tensile strength in the first mentioned alloy, cold reduction of 45 per cent is necessary. The accompanying elongation is 4 per cent and proof stress is 86,000 pounds

per square inch. For the second alloy, cold reduction of only 15 per cent gives the tensile strength of 161,000 pounds per square inch, elongation of 12.5 per cent but proof stress of only 52,000 pounds per square inch.

Thus in selecting the desired tensile strength, providing the proof stress is of importance—it is necessary to consider the amount of cold work needed to produce this tensile strength, since the values for proof stress are dependent upon per cent cold work. In most of

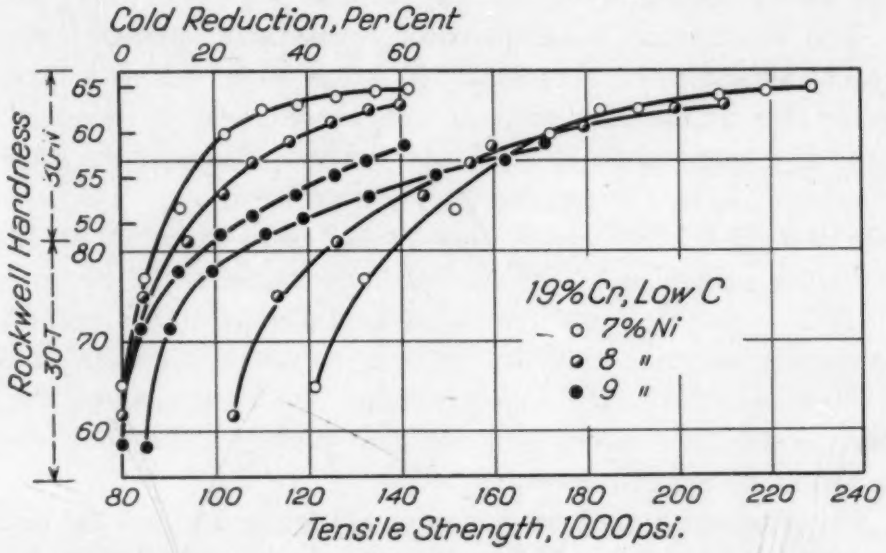

Fig. 25—Same type of Summary as that Shown in Fig. 21, but for Alloys Containing About 0.05 Per Cent Carbon, 19 Per Cent Chromium, and 7 to 9 Per Cent Nickel.

the alloys studied, 30 per cent cold reduction was necessary to obtain a proof stress equal to or better than 50 per cent of the tensile strength.

Clearly then, the proof stress values are a function of the degree of "metastability" of the alloys. The latter is determined by the analysis and rather easily affected by such processes as cold working. The gross effect of changes necessarily induced on the way toward equilibrium, as expressed by the mechanical properties of the alloys, is determined by the complex and interdependent factors. The influence of one single alloying element, chromium, nickel, or carbon cannot be extrapolated: this is the conclusion which we have already stated.

Various circumstances often preclude the possibility of adequate testing of materials, especially in production. Time honored "short cuts" are of necessity employed. Interdependence of tensile strength

and hardness is frequently accepted and in many cases not without justification. Our studies of austenitic and pseudo-austenitic alloys convinced us that generally accepted postulates do not necessarily apply to these alloys. It appeared desirable, therefore, to look into the matter of hardness, and, indirectly, of hardenability. A rather complete picture can be obtained when hardness of the cold-rolled alloys is plotted against (a) degree of cold work and (b) the tensile strength (after cold work). Experimental results for high carbon alloys are assembled in Fig. 21, 22, and 23.

The results are self-explanatory and their detailed discussion does not appear to be necessary. We may point out as a matter of interest that as the sum total of alloying element is increased, and the nickel content is above 7 per cent, various alloys of the same tensile strength have correspondingly similar hardness. To illustrate: 17-9, 19-8, 19-9, 18-8, 18-9 alloys with tensile strength of 125,000 pounds per square inch all have hardness values of 83-85 on Rockwell 30T scale.[12] With tensile strength of 160,000 pounds per square inch, corresponding hardness is between 58.5-60.0 on Rockwell 30N scale, etc. Strangely enough the minimum of the sum total (per cent chromium plus per cent nickel in the alloy) needed seems to be 26 per cent.

For alloys of higher chromium and 7 per cent nickel the relationship between hardness and tensile strength does not hold. Neither does it hold for alloys with low carbon (0.05-0.06 per cent), regardless of the amount of alloying elements present. In Figs. 24 and 25 are plotted the results characteristic of such alloys. For a given hardness, for example of 80 on "Rockwell 30T" scale the corresponding tensile strength will vary between 105,000 and 145,000 pounds per square inch. It will be noted, however, that after heavy reduction by cold working, tensile strength and hardness become interrelated. It should have been stated that this observation applies to every alloy investigated, regardless of its composition. We should like to mention again that, apparently, each alloy has a certain, full hardening capacity.

Part III

At the very beginning of this communication the reference was made to properties and characteristics of the alloys under discussion.

[12]Rockwell 30T and 30N hardness scale were used because they yield more reliable results on thin gage samples.

The word "characteristics" was used to denote such properties as, for example, bending and forming ability, which are so important in engineering work yet so very difficult to estimate quantitatively. To illustrate our contention, we refer the reader to Table V in which are given tensile strength, ductility as measured by elongation, and the smallest radius over which the cold-worked material will bend flat on itself (parallel to the direction of rolling) without cracking.

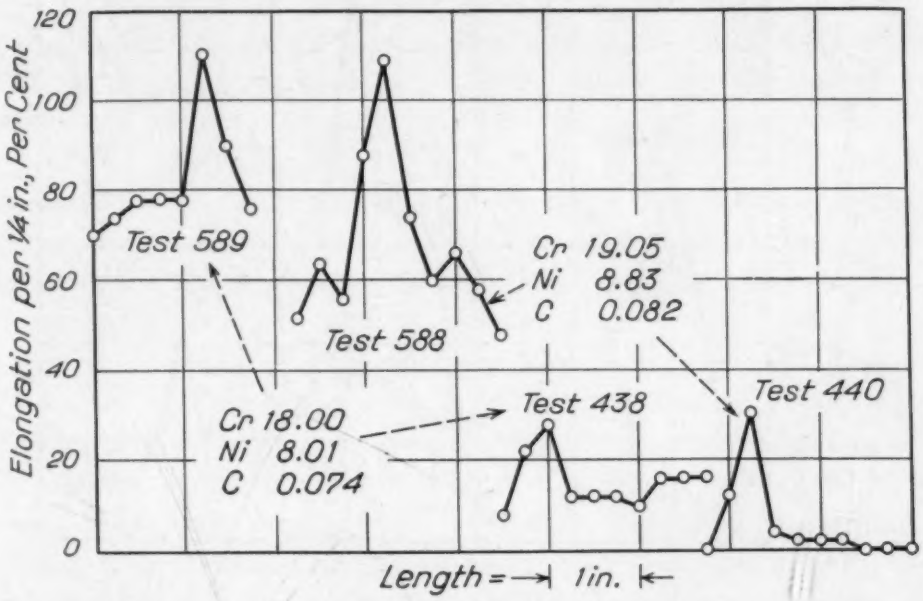

Fig. 26—Distribution of Elongation in Each Quarter Inch Over the 2-Inch Gage Length for Alloys as Annealed (Tests 588 and 589) and After Cold Rolling to About 170,000 Pounds Per Square Inch (Tests 438 and 440).

In Table V the alloys are grouped according, first, to their composition, and, second, to tensile strength and elongation. To complete the table, the gages of the tested specimens are also included. Before discussing this table, we wish to state that we do not possess, as yet, accurate knowledge regarding the influence of relatively small variations in thickness upon bending and forming habits of the alloys of the same mechanical properties. Therefore, that part of the table which contains information on bending should be considered as only qualitative.

While greater elongation (for a given tensile strength and regardless of the gage tested) is generally accepted to mean better bending quality, the relationship is by no means a simple one. It would be difficult to predict that alloys with 20 and 25 per cent elongation (alloys 7 and 26) or 11.0 and 14.0 per cent (alloys 9 and 10) would

have altogether different bending properties. It will be noted, as a general observation, that for a given tensile strength and chromium-nickel content, alloys with higher carbon showed superior bending properties. If we disregard the composition and compare the elongation with bending, the results are controversial; some alloys with the same elongation (number 10 and 28 practically the same gage)

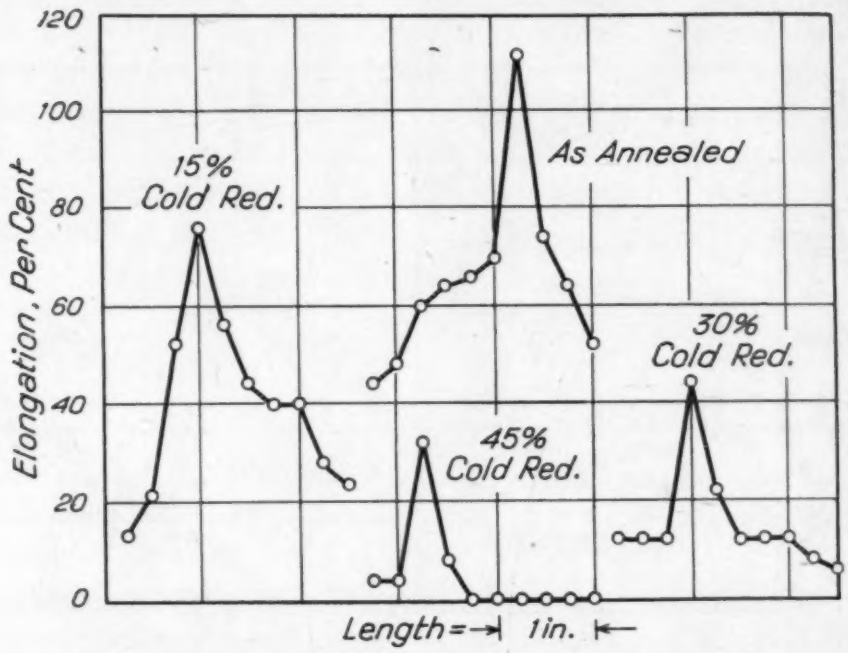

Fig. 27—Distribution of Elongation in Each Quarter Inch Over the 2-Inch Gage Length for an Alloy Containing 0.05 Per Cent Carbon, 18.22 Per Cent Chromium, and 9.14 Per Cent Nickel, as Annealed, and After 15, 30, and 45 Per Cent Cold Reduction.

have the same bending characteristic, while others (alloys number 15 and 17, also 7 and 29) differ in their forming and bending ability. Thus, the ability to bend or form is not so much a function of tensile strength (within reasonable limits), but is primarily governed by the composition, carbon being the outstanding factor as has already been stated.

The initial, that is, in the fully softened state, characteristics of the alloys must be the important, if not the predominant, factor. To ascertain this factor or influence and to elucidate upon the data contained in Table V, further experimental work was necessary.

Tensile samples, of the same thickness, were most carefully marked with very fine lines 0.25 inch apart. After the tensile test was completed and specimen broken, elongation per 0.25 inch was measured and the distribution of elongation over the length of 2½

inches was ascertained. The results are to be found in Figs. 26, 27, 28 and 29 and will be discussed now.

The first in the series of the experiment is Fig. 26. Two alloys of the most generally used composition, (namely 18 chromium, 8.01 nickel, and 0.07 carbon; 19.05 chromium, 8.83 nickel and 0.08 carbon) have, after full anneal, the tensile strength of, in round figures, 102,000-104,000 and 90,000-95,000 pounds per square inch, respectively. The difference being not very great, indeed. When cold-rolled, starting and finishing with the same gage, the respective tensile strength and elongations were found to be 175,000 pounds per square inch, 16 per cent and 165,000 pounds per square inch, 6.0 per cent, for about 45 per cent reduction. For the purpose of our discussion, the cold-rolled strengths are quite comparable. Both alloys are characterized, in the fully softened state, by very high and quite comparable elongation—82 and 77 per cent in 2 inches. After cold rolling not only the actual values but also the distribution of the elongation is altogether different. In alloy 19-9 (test 440, Fig. 26) the ductility of the metal at the break is equal to that of 18-8 (test 438, Fig. 26) but only one quarter of an inch, each side, from the place of rupture the metal has been distorted to a negligible degree. On the other hand as much as 16 per cent elongation was found in the alloy 18-8 (test 438) one inch and a half from the place of rupture. Just what is the process that takes place during the cold deformation of these alloys, to which we ascribed, in our opinion with full justification, various degrees of stability? An observed phenomenon, which we hope in the future communication to show by means of a film, but, in the paper shall attempt to describe, leads us to the formulation of certain ideas. The observation was as follows:

In alloys with high nickel—the type we previously referred to as "more stable"—and in which the elongation is confined to a narrow area, the deformation starts at a certain point, a limited number of slip lines appear. Then, the sample begins to "neck-down" and finally ruptures. In low nickel alloys—with more even distribution of elongation—the appearance of the first slip lines is followed by a number of others, alongside the first, giving the impression that the slip lines run away from the location where they first started. Finally the whole of the sample is covered with slip markings from one end to the other and only then necking begins. Incidentally, the necking (and finally breaking) may or may not occur at the spot where the first slip lines were noticed.

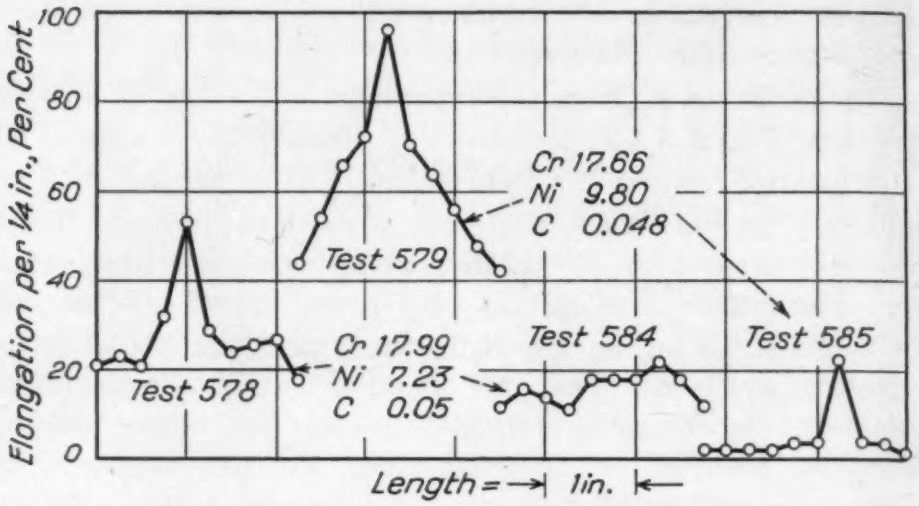

Fig. 28—Distribution of Elongation in Each Quarter Inch of the 2-Inch Gage Length for Alloys as Annealed (Tests 578 and 579) and After Cold Rolling to About 165,000 Pounds Per Square Inch (Tests 584 and 585).

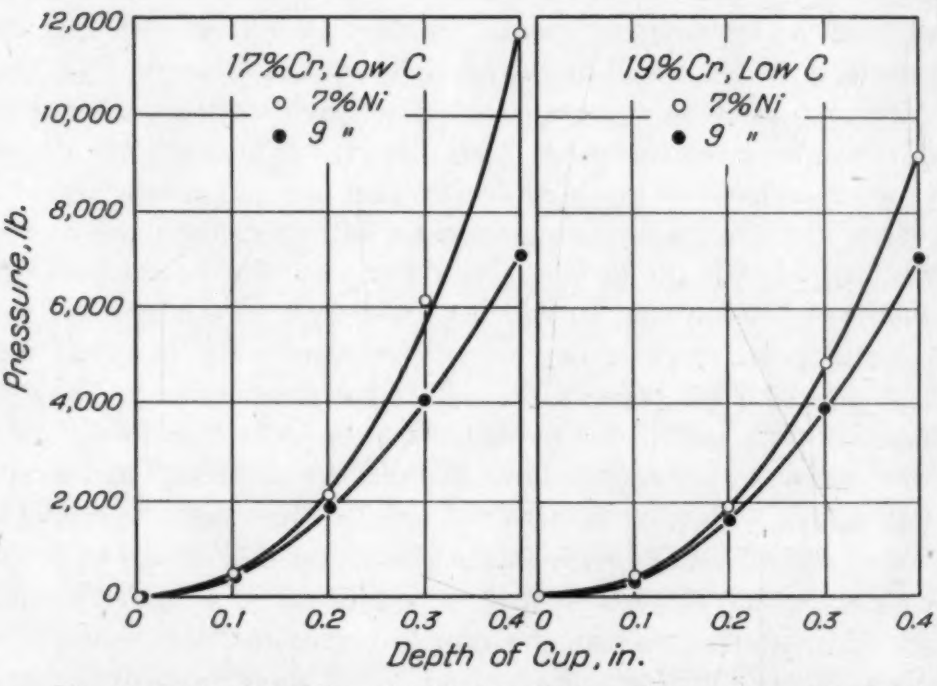

Fig. 29—Load on the Olsen Machine Required to Form a Cup of the Depth Shown, in a Sheet, 0.038 Inch Thick, of the Indicated Analysis.

We explain the observation as follows. At the very first moment of distortion, when first lines appear and when infinitesimally small "necking" or reduction in cross section of the sample takes place, two phenomena take place. One, shall we call it (a) is the increase in tensile strength of the material in pounds per square inch due to de-

composition induced by cold deformation at the section where slip lines formed and the infinitesimally small "necking" occurred; another (b) is the increase in total load upon cross section of the sample under test due to the reduced area at the point of "necking." If (a) is greater than (b) the metal is stronger at the point of first permanent distortion and any further slip will, necessarily, take place in the metal adjacent to the "hardened" portion until the whole sample becomes distorted and finally ruptures. If (a) is less than (b) necking, once begun, will continue and final break will occur where first slip started with practically no deformation in the metal even a short distance away from the actual break.

If, during the process of cold working—and because of it—the full extent of hardenability through decomposition has been exhausted the phenomenon as described under (a) above will not take place while the metal is further deformed either by forming, bending or for that matter testing in the machine. In the latter case, the per cent total elongation will be found quite low. The characteristics of an alloy of such a degree of cold deformation should not be dissimilar to those of cold-worked "pure" metals or alloys that do not exhibit decomposition upon cold working. Since this paper is not intended to be a discussion of many interesting phenomena found in cold-worked metallic substances we do not feel justified in going any further than merely suggesting a comparison. However, this comparison should not be accepted too literally.

Following this thought, another set of experiments has been performed. An alloy containing 0.05 per cent carbon, 18.22 per cent chromium, and 9.14 per cent nickel was cold-rolled 15, 30 and 45 per cent of the total reduction. Samples were secured after each degree of cold work, tested in accordance with the previously outlined method and the results recorded in Fig. 27. Of interest to us are the results after 30 and 45 per cent cold deformation. The former shows 44 per cent elongation at the rupture and minimum of 12 per cent elsewhere, except for the last ¼ inch at one extreme end. The 45 per cent reduction sample shows 32 per cent elongation in the ruptured section, very little in two adjoining sections, one on each side, and none in the rest of the sample. The 12 per cent represents the extent of elongation possible prior to hardening of the alloys to the full capacity. Measurements on many samples strongly suggested that *fully worked* alloys possess a characteristic elongation at the break, the value of such elongation being dependent upon the com-

position of the alloy,[13] averaging from 15 to 35 per cent. It was further found that, in alloys of the same carbon content, the excess elongation over that at the break of fully worked material corresponds to, or, shall we say, serves as an indicator of the bending and presumably forming characteristics. It is especially true of high carbon alloys. In the low carbon group a few exceptions were noted. And since, as was just emphasized, the characteristic elongation is different for different alloys it is not surprising to find contradictory, as would appear on the surface, observations as recorded in Table V below.

Table V

Alloy No.	C	Cr	Ni	Tensile Strength Pounds Per Square Inch	Elongation in 2 Inches	Gage	Radius in Inches
7	0.05	17.00	8.05	150,000	20.0	0.031	0.070
26	0.14	16.80	8.15	150,000	25.0	0.028	0.032
9	0.05	16.83	9.79	150,000	11.0	0.022	0.070
10	0.13	16.90	9.75	150,000	14.0	0.024	0.032
13	0.05	17.62	8.51	150,000	23.0	0.026	0.050
27	0.14	17.95	7.90	150,000	25.0	0.027	0.032
15	0.05	17.66	9.80	175,000	3.0	0.018	0.070
14	0.13	17.65	8.60	175,000	8.0	0.019	0.032
17	0.06	18.68	7.33	175,000	11.0	0.0285	0.070
28	0.16	19.70	6.75	175,000	15.0	0.022	0.032
17	0.06	18.68	7.33	200,000	4.0	0.023	0.090*
28	0.16	19.70	6.75	200,000	7.0	0.018	0.050
7	0.05	17.00	8.05	150,000	20.0	0.030	0.070
29	0.16	18.68	7.90	150,000	22.0	0.026	0.032

*Broke.

As was demonstrated in previous pages, a predetermined tensile strength can be obtained in various alloys by subjecting the latter to a different amount of cold reduction. The amount of necessary cold reduction is governed by the composition, that is to say, by the properties of the alloy in the fully softened condition. Thus, the selection of the material for the desired tensile strength and ductility after cold rolling should be governed by the considerations just described. It is rather well illustrated by Fig. 28 in which alloys showing superior ductility in the annealed condition are not the proper selection for the desired tensile strength and a certain amount of ductility, after cold working.

It is to be clearly understood by the patient reader that the above-discussed studies related to the property of "workability," or

[13]The influence of gage must of course be considered. In the case under discussion measurements were made on the samples of the same gage.

whatever may be the name for it, are only preliminary. It is, indeed, realized that the problem is very complex. The mere ability to bend or to form is not all. It should preferably be accompanied by the arbitrary property of "ease" or "difficulty" in actual forming operation. To study the latter problem it was thought wise to assemble some quantitative data, and to do this an Olsen tester was employed. In-

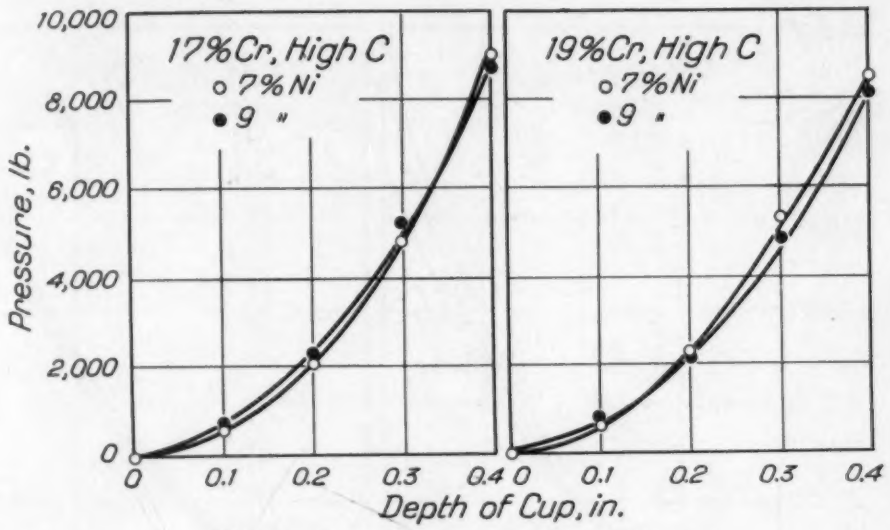

Fig. 30—Same Type of Summary as Fig. 29.

stead, however, of testing samples to destruction, careful readings were taken of the pressure in pounds necessary to draw a cup of a certain depth, namely, 0.1, 0.2, 0.3 and 0.4 inch. Fig. 29 shows the comparative values for (a) low carbon, seventeen per cent chromium alloy and (b) low carbon, nineteen per cent chromium alloy, in both cases varied in nickel. In the first series an increase in nickel from 7 to 9 per cent resulted in the decrease of required power from 11,700 pounds to 7100 pounds, or approximately 40 per cent. In the second series from 9200 to 7000 pounds or about 23 per cent. This pronounced influence of nickel could have been predicted on the basis of the herein discussed "instability" of low carbon 17-7 alloys as shown by comparing Fig. 29 and Fig. 30, the latter pertaining to alloys of high carbon content. It will be seen that increase in carbon content nullifies the influence of nickel (as noted in low carbon alloys) and gives to the alloys practically the same characteristics. The necessary pressure is higher than that required by the low carbon high nickel alloys.

The same influence of carbon will be observed in those alloys (Fig. 31) in which chromium is a variable.

From the same curves it can also be concluded that the influence of chromium is least of all.

These statements are, of necessity, of a general nature and space does not permit the reproduction of all available data.

This discussion has been confined, purposely, to the systematic study of the alloys with carbon content at the extreme limits of gen-

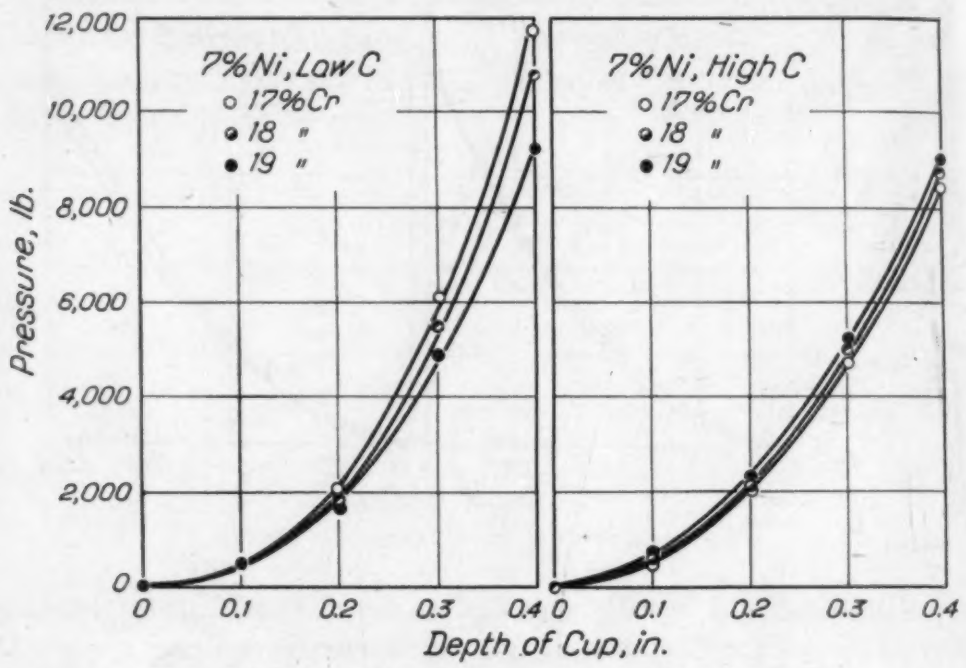

Fig. 31—Same Type of Summary as Fig. 29.

erally used specifications. Similar study on alloys with intermediate carbon (0.09-0.10 per cent) will have to be communicated later.

As a sort of conclusion, we include Table VI. It is hoped that the progressive engineer, or designer, will find in it the data that he needs.

The very name "stainless" implies the responsibility of the studied alloys toward corrosion. Because of the relatively wide limits of alloying elements (chromium and nickel) and of carbon included in the above summarized work, the resistance to corrosion naturally varies. These variations estimated by known and generally adopted methods have not been found large enough to disqualify any of the mentioned alloys as "stainless." The gathered data is so voluminous that it will have to be made the subject of another communication.

Table VI

Properties Attainable in Alloys of Various Composition

Per Cent C	Per Cent Cr	Per Cent Ni	Elongation in 2 Inches	Gage	Smallest Radius Over Which Bend Was Satisfactory—Inch
Cold-Rolled to a Tensile Strength of 150,000 pounds per square inch. Starting with same gage (0.038 inch) annealed					
0.10	15.66	7.46	32.0	0.036	0.032
0.13	15.66	7.62	40.0	0.034	0.032
0.05	17.25	7.07	15.0	0.034	0.050
0.10	17.05	7.01	45.0	0.034	0.032
0.14	17.20	7.18	53.0	0.036	0.032
0.14	17.98	7.04	51.0	0.034	0.032
0.13	17.65	8.60	19.0	0.025	0.032
0.05	18.63	8.17	19.0	0.027	0.050
0.05	16.83	9.79	7.0	0.021	0.070
0.05	17.66	9.80	10.0	0.024	0.050
0.05	18.22	9.14	5.0	0.020	0.050
Cold-Rolled to a Tensile Strength of 175,000 pounds per square inch.					
0.13	16.47	6.60	34.0	0.034	0.050
0.10	15.66	7.46	23.0	0.032	0.050
0.13	15.66	7.62	27.0	0.030	0.032
0.05	17.25	7.07	9.0	0.027	0.090
0.14	17.20	7.18	35.0	0.032	0.032
0.05	16.83	9.79	2.0	0.017	0.070
0.14	17.98	7.04	34.0	0.029	0.032
0.05	18.63	8.17	7.0	0.023	0.050
0.05	18.22	9.14	3.0	0.019	0.090
Cold-Rolled to a Tensile Strength of 200,000 pounds per square inch.					
0.13	16.47	6.60	27.0	0.030	0.050
0.10	15.66	7.46	15.0	0.030	0.090
0.13	15.66	7.62	19.0	0.024	0.032
0.14	17.98	7.04	22.0	0.027	0.032
0.06	18.68	7.33	4.0	0.021	0.090
0.16	19.70	6.75	7.5	0.021	0.050
Cold-Rolled to a Tensile Strength of 225,000 pounds per square inch.					
0.13	16.47	6.60	20.0	0.025	0.050
0.14	17.20	7.18	12.0	0.023	0.090
0.14	17.98	7.04	12.0	0.022	0.070
0.06	18.68	7.33	2.5	0.016	0.050

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DISCUSSION

Written Discussion: By Paul Ffield, materials engineer, Bethlehem Shipbuilding Corporation, Quincy, Mass.

In my former position as metallurgical engineer for the Goodyear-Zeppelin Corp., I was called upon to investigate the use of stainless steels in airship construction. This investigation started in 1929 and carried over a period of six years and was prompted by the fact that we were accused of being behind the times in using strong aluminum alloys instead of stainless steel in our structures. Our investigation was quite comprehensive and we encountered many difficulties which could have been avoided if Dr. Krivobok had conducted this excellent research about six years sooner.

In investigating aluminum alloy structures we had developed both optical and autographic extensometers. The latter would plot the stress-strain curve at magnifications up to × 1000. When we tested high tensile 18-8 with this extensometer we learned that what might be termed the engineering characteristics of the alloy were very different from the sales version of the tensile properties of the material.

We encountered proportional limits as low as 5000 pounds per square inch on 200,000 pounds per square inch material. The elastic modulus would vary on different brands of 18-8 from 22,000,000 to 30,000,000 and we would get wide variations in elongation even though the tensile strength remained constant. Dr. Krivobok and his associates have cleared my mind on a number of such points which six years ago were baffling to me as an engineer.

My experience in fabricating experimental structures of high tensile 18-8 indicates that variations in properties occur from sheet to sheet of the material. There still appears to be some factor outside the analysis in either the heat treatment or rolling of the material which will produce variations in tensile properties from the same heat of steel. I look for enlightenment on this point at some time in the future by the authors.

Concerning proof stress there is a method by raising the proof stress other than by cold rolling which should not be overlooked when studying the structural applications of austenitic alloys. The method was to preload the material above the yield point thereby raising its yield strength to a new level. It so happens that the duralumin girders and steel tie rods in the airship HINDENBURG have in some cases been so treated. This was accomplished by either stretching the sheet after rolling or as in the case of the HINDENBURG, stretching the girder booms after fabrication. This latter method has the advantage of reorienting the locked up shop fabrication stresses.

I would be interested to know whether the authors tested their specimens parallel to, or perpendicular to the direction of rolling. My Goodyear-Zeppelin experience indicated that a slightly higher elastic modulus and proof stress were obtained when transverse specimens were pulled. However, the elongation would be proportionately lower.

I wish to congratulate the authors on this excellent paper which I believe is the first attempt to furnish the designing engineer with the comprehensive information which he needs.

Written Discussion: By T. F. Olt, supervising metallurgist, and R. S. Burns, research engineer, American Rolling Mill Co., Middletown, Ohio.

The authors are to be congratulated on their splendid contribution to the literature covering austenitic stainless steels of the chromium-nickel variety. In general, the data presented are in agreement with those obtained in our laboratory on samples of varying chromium and nickel analysis selected from commercial heats. At the time, we undertook a study of the factors affecting the work-hardening rate of austenitic stainless steels because of the tremendous variation in results encountered in the commercial production of high tensile stainless. It may well be stated at the outset that the manufacture of high tensile stainless steel requires that the specified thickness, minimum tensile strength, yield strength, ductility, the proper degree of flatness, and other desirable attributes must all be produced simultaneously.

Considering high tensile stainless of, say, 150,000 pounds per square inch, which no doubt enjoys the widest usage at the present time, the authors in the data presented point out that a cold reduction (final) of from 8 to 45 per cent (roughly), depending on the carbon-chromium-nickel relationship, is required. That such a tensile strength can be produced without any final cold reduction whatever (or in the "dead soft annealed" condition) by further variations in composition is shown in Table A.

These materials had been hot-rolled to 0.100 inch, annealed at 2100 degrees Fahr. for 5 minutes plus air-cool, pickled, and cold reduced to 0.055 inch and reannealed at 2100 degrees Fahr. for 5 minutes, pickled, and cold reduced to 0.031 inch and again reannealed at 2100 degrees Fahr. for 5 minutes before pickling and testing. It will be noted that for a given chromium and nickel content, also for a given tensile strength, the higher carbon material has the better percentage of elongation. The complete range of corrosion resistance of these alloys has not been studied by us; however, all of the alloys are quite resistant to a 20 per cent salt spray, and no doubt would give sufficient resistance in atmospheric exposures to come within the stainless or rustless classification.

The yield strengths[1] shown in Table A are probably too low to consider using materials of the analyses described, in the fabrication of high strength light weight structures; however, they are all in the general range of yield strength obtained in the austenitic stainless or rustless alloys that have not been cold-worked as a final operation. The authors have pointed out that the only way to raise the proof stress (also yield strength) an extensive amount is through cold work. The increase in yield strength is governed much more by the amount of cold reduction than by variations in analysis, although varia-

[1]The yield strength which we have specified in Table A at 0.5 per cent elongation under load is often specified at 0.2 per cent set E = 25,000,000 or for 150,000 pounds per square inch tensile at 0.0064 inch strain under load, and may be specified many other ways. The proof stress is always a lower value than 0.2 per cent set E = 25,000,000. It may be higher or lower than 0.5 per cent elongation under load depending on the amount of cold reduction. Fig. B shows the relationship between 1 per cent yield strength and 0.2 per cent set E = 25,000,000 yield strength. It may be noted that the relationship is within a variation of ±5 per cent of the mean curve on the three different analyses shown. The determination of the 1 per cent yield strength is quite accurate and very simple on any production laboratory testing machine without the use of expensive and delicate extensometers, while the determination of the 0.2 per cent set E = 25,000,000 yield strength, being on the steeper part of the load deformation curve, requires the use of more accurate and delicate extensometers and manipulation to obtain a reasonably accurate load value.

Table A
Effect of Composition on Physical Properties of Chromium-Nickel Alloys

Sample	Composition of Alloy C	Cr	Ni	Strength Lb. Per Sq.In. Yield*	Tensile	Elongation Per Cent in 2 Inches	Rockwell Hardness	YS/TS
5	0.06	17.5	6.2	30,300	153,000	22.9	B-90	0.195
2	0.07	15.8	7.2	31,300	149,500	21.5	B-90	0.209
3	0.06	16.6	6.3	37,700	170,100	13.5	C-24	0.222−
1	0.06	15.8	6.4	47,900	181,000	9.0	C-32	0.265+
H5	0.12	16.9	6.0	41,400	157,700	32.1	B-95	0.261−
H8	0.12	15.6	6.1	46,400	187,600	17.0	C-28	0.246+

*Yield strength at 0.5 per cent elongation under load, rate of strain 0.05 inch per minute to yield strength, 0.2 inch per minute to break (head speed of testing machine ½ inch per minute from yield strength to break).

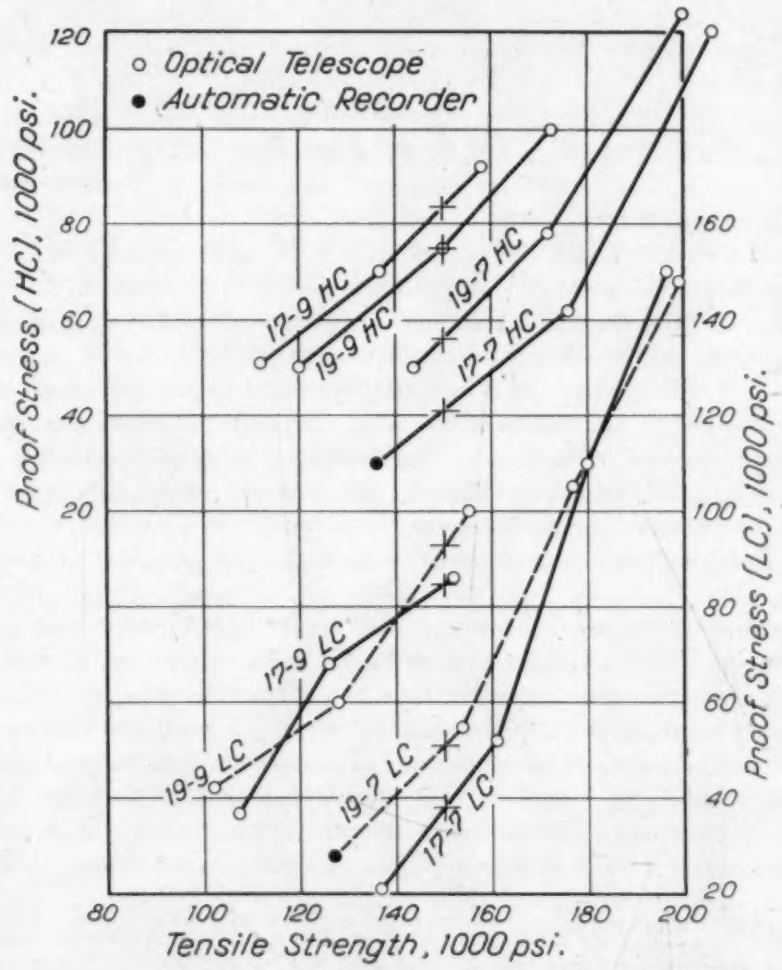

Fig. A

tion in analysis does have an effect. This is shown by the data given in Table IV, "Values for Proof Stress," which we have taken the liberty to plot against tensile strength in Fig. A. The proof stress at 150,000 pounds per square inch tensile strength varies with analysis according to Table B.

Table B
Effect of Analysis on Proof Stress and Per Cent of Elongation at 150,000 Pounds Per Square Inch Tensile Strength

Type Alloy	Analysis C	Analysis Cr	Analysis Ni	Proof Stress*	Per Cent Elongation in 2 In.**	Reference
17-7 LC	0.053	17.13	7.07	38,000	17.0	Fig. 2
17-9 LC	0.05	16.83	9.79	85,000	10.0	"
19-7 LC	0.056	18.68	7.33	51,000	23.0	Fig. 4
19-9 LC	0.051	18.22	9.14	92,500	8.0	"
17-7 HC	0.14	17.26	7.07	40,500	56.0	Fig. 11
17-9 HC	0.13	17.08	9.66	83,500	14.0	"
19-7 HC	0.16	19.68	6.68	56,000	28.0	Fig. 13
19-9 HC	0.16	18.88	8.78	75,000	16.0	"

*In scaling off these values, it was assumed that the variation in proof stress with increasing cold reduction was linear, which is not true. However, the errors introduced are minor as compared to the tremendous difference engendered by varying the nickel content as most of the points are scaled off close to a determined value.

**The percentage elongation values have been obtained by scaling the curve in the figure indicated at the Per Cent Cold Reduction necessary to produce 150,000 pounds per square inch tensile strength on that particular composition.

It can be seen readily that with 7 per cent nickel, either high or low carbon and chromium, the proof stress lies between 38,000 and 56,000 pounds per square inch when cold reduced to 150,000 pounds per square inch tensile

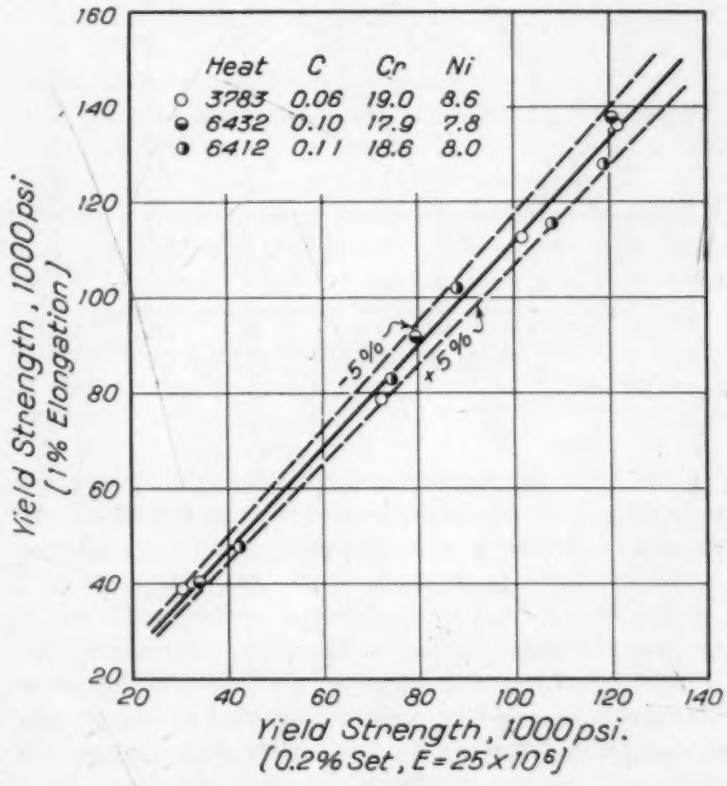

Fig. B

strength; while with 9 per cent nickel, the proof stress range is from 75,000 to 92,500 pounds per square inch for the same tensile strength. We note that the data in Table IV do not check with the data plotted in Figs. 2, 4, 11, and

13, especially with respect to per cent elongation in the tensile test. For instance, in Table IV, the 17-7 HC as annealed shows 65 per cent elongation and a tensile strength of 136,000 pounds per square inch, whereas in Fig. 11 this same material as annealed shows an elongation of 80 per cent in 2 inches and a tensile strength of 130,000 pounds per square inch. Also, the 17-9 HC shows 114,000 pounds per square inch tensile strength and 80 per cent elongation in Table IV, but 105,000 pounds per square inch tensile strength and 71 per cent elongation in 2 inches in Fig. 11. These differences in tensile strength are

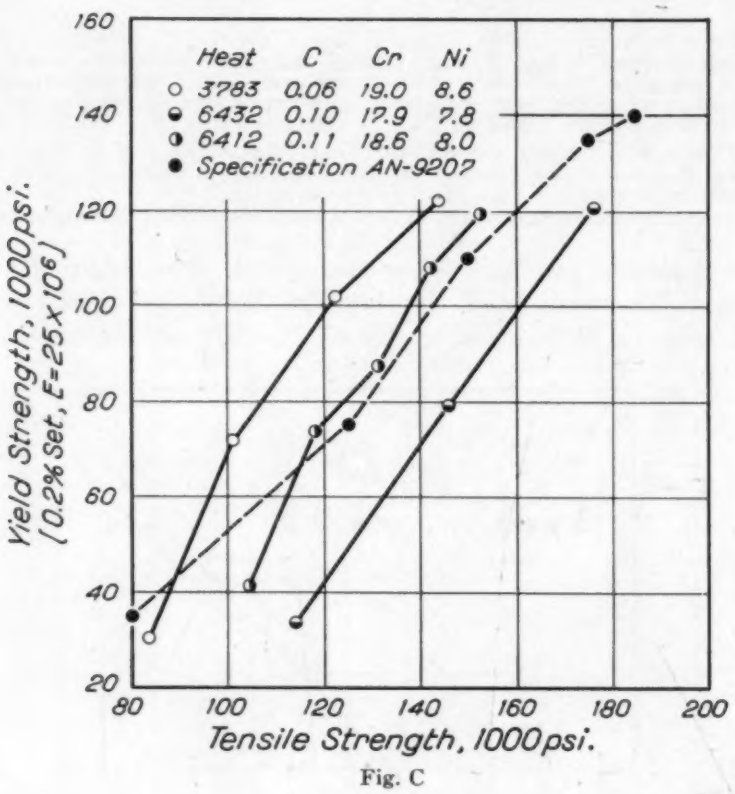

Fig. C

possibly within the limits of experimental error, but the differences in ductility can hardly be explained on this basis, inasmuch as in one case Table IV shows a higher value and in the other case the figure shows the higher value.

As the authors point out, in many cases the yield strength specification requires the judicious selection of the proper analysis and cold reduction in order that the requirements of the specification can be fulfilled. Just how judicious this selection must be is shown by Fig. C, which shows the relationship between analysis, yield strength as specified in Government Specification AN-9207, and tensile strength on three heats with analyses not too unlike to be considered well within a commercial melting range. It can be seen readily that Heat 6432 lies to the right of the specification line for all values of tensile strength. This heat analyzes 0.10 per cent carbon, 17.9 per cent chromium, and 7.8 per cent nickel, well within the specified analysis range of 0.12 per cent carbon maximum, 17 per cent chromium minimum, and 7 per

cent nickel minimum. This heat, when the tensile strength has been raised to 150,000 pounds per square inch through cold work, will meet all requirements except the yield strength (specified at 0.2 per cent set $E = 25 \times 10^6$). In order to obtain the specified yield strength for 150,000 pounds per square inch minimum tensile strength on this heat, it would be necessary to raise the tensile strength to approximately 170,000 pounds per square inch instead of the 150,000 pounds per square inch tensile strength as specified and concomitantly reduce the ductility and bending qualities. In other words, high yield strength and decreased workability, or lowered yield strength and better fabricating qualities can be considered as going hand in hand. A comparison between Heats 6432 and 6412 in Fig. C will adequately show just how small a variation in analysis will produce success or failure in meeting a given specification.

If we have dwelled too long on the relationship between analysis and yield strength, tensile strength, ductility, and cold working characteristics, which the authors have shown so well in their paper, it is merely to again emphasize these important relations for consideration in the preparation of specifications for and applications of this class of material, for which we beg the authors' kind indulgence.

Written Discussion: By Russell Franks, research metallurgist, Union Carbide and Carbon Research Laboratories, Inc., Niagara Falls, New York.

This paper by Dr. Krivobok and Mr. Lincoln contains an enormous quantity of valuable data relating to the characteristics of chromium-nickel steels of high tensile strength resulting from cold working. The data are especially pertinent at present because of the interest shown by those desiring to employ the 18-8 steels in light weight high strength construction. While the use of the steels in this connection has expanded rapidly because of the excellent results already attained, some difficulties have been met with, thereby indicating the necessity for a thorough study of the characteristics of the chromium-nickel steels in the cold-worked condition. The utility of construction of this type undoubtedly depends upon the stability of the system as a whole, which is partly controlled by engineering design; to what extent this stability depends upon the stableness of the austenite comprising the 18-8 steel employed is a question of considerable importance.

It is interesting to learn that within certain limits the elements carbon and nickel have a greater effect than chromium in producing austenitic chromium-nickel steels capable of being cold-worked to give excessively high tensile characteristics. It is brought out by Dr. Krivobok and Mr. Lincoln that with a given amount of cold work the higher strengths are developed in the less stable steels. These steels are apparently capable of developing higher strength during cold working because of the readiness with which the austenitic condition can be altered. It is also brought out in the paper that in addition to the higher strength, the less stable chromium-nickel steels exhibit in many instances greater elongation (in the 2-inch gage length) than the more stable 18-8 type steel which must be cold-worked to a greater degree to give approximately the same strength. We should probably reason from this that the less stable 18-8 steels are the more suitable in high strength applications, but let us analyze the problem a little further. Dr. Krivobok and Mr. Lincoln in-

geniously show that if the 2-inch gage length is divided into a number of ¼-inch sections, during test a greater amount of elongation will take place in some one of these ¼-inch sections than in any of the others. They further reveal that the elongation obtained in the unstable steels is more evenly distributed over the 2-inch gage length than is the case of the austenitic chromium-nickel steels of greater stability. In the latter steels the elongation during fracture is quite pronounced in one of the ¼-inch gage lengths within the 2-inch section. This indicates that when the more stable steels fracture, a greater reduction in area is obtained, and we are wondering why this should not be considered advantageous as well as the amount of elongation that occurs simultaneously within the 2-inch gage length.

Stability is an extremely important consideration and the extent to which this is varied to obtain high strength is equally important. In other words, how far can the increase in strength be carried by cold rolling without seriously affecting other desirable properties? In order to illustrate the point we should like to cite the following results: A cold-rolled steel containing 18.95 per cent chromium, 7.70 per cent nickel, and 0.07 per cent carbon exhibited in a standard tensile test using a 0.505-inch section, a maximum strength of 146,000 pounds per square inch, a yield point of 135,900 pounds per square inch, an elongation of 20 per cent in 2 inches and a reduction in area of 61 per cent. The steel also exhibited an Izod impact value of 34 foot-pounds. In another test a more stable steel containing 17.42 per cent chromium, 8.24 per cent nickel and 0.06 per cent carbon exhibited after cold rolling a maximum strength of 138,000 pounds per square inch, a yield point of 128,300 pounds per square inch, an elongation in 2 inches of 29 per cent and a reduction in area of 68 per cent with an Izod impact value of 67 foot-pounds. While these results may not be entirely comparable to those obtained in cold-rolled strip, they do show that after cold working to approximately the same degree the higher strength steel is inferior in toughness to the more stable steel with a somewhat lower strength.

Another important point in connection with the high strength 18-8 steels has to do with fatigue, and even though Dr. Krivobok has not included data of this nature in the present publication we sincerely hope that this property will be considered. The data obtained may inform us of the most useful composition, and the extent to which the strength of this composition should be increased by cold rolling to obtain optimum service.

There is a possibility that many of the light weight high strength structures being used at present will be employed at temperatures decidedly below normal, particularly when equipment is shifted for service in different parts of the country. While all the published data and experience so far have indicated that the 18-8 steels have remarkably good properties at low temperatures there are indications that if too much ferrite is present their toughness may be detrimentally affected. Thus, it seems important that the low temperature characteristics of cold-rolled strip of these steels should be investigated with the thought that the data obtained would aid in designing the most appropriate steel for such applications.

We congratulate Dr. Krivobok and his associates upon the work they

have already done, and it is hoped that the work will be continued because the data available on this subject are far from complete.

Written Discussion: By Joseph Winlock, chief metallurgist, Edward G. Budd Mfg. Co., Philadelphia.

I have read with a great deal of pleasure the very interesting and timely paper by Dr. Krivobok, Lincoln and Patterson on the properties of the austenitic stainless steels after cold rolling. To those interested in the use of these steels which are so valuable for structural work in which the weight-strength ratio is of great importance, Dr. Krivobok's paper furnishes in orderly and simple fashion the results of a very extended research. Only a careful reading can give an idea of the enormous amount of work which he and his associates have done.

Dr. Krivobok's experiments check the results of the experience we have had in our shop in using these alloys for light weight structures, namely, that the low nickel high carbon austenitic stainless steels yield a more ductile steel after cold rolling than when the relative amounts of those alloys in the metal are reversed.

In this connection, the following table is of interest. This table shows the tensile properties after cold rolling to a minimum tensile strength of 150,000 pounds per square inch and a maximum tensile strength of 165,000 pounds per square inch, together with the percentage reduction by cold rolling. A range of tensile strengths of this magnitude is obviously necessary when it is considered that in practice gage tolerances within very close limits as well as tensile strength must be obtained. The data for this table were obtained from Figs. 3 to 5 and 11 to 13 in the paper.

			Pounds Per Square Inch		
			150,000	165,000	Cold Roll
Per Cent Chromium	Per Cent Nickel	Per Cent Carbon	Per Cent Elongation	Per Cent Elongation	Per Cent Reduction
17.0	7.0	0.05	18.0	12.0	5-15
	8.0		20.0	12.0	20-30
	9.0		10.0	7.0	42-52
18.0	7.0	0.05	19.0	12.0	10-20
	8.0		24.0	12.0	30-40
	9.0		12.0	7.0	38-48
19.0	7.0	0.05	22.0	18.0	10-22
	8.0		18.0	12.0	28-35
	9.0		8.0	5.0	45-55
17.0	7.0	0.14	60.0	42.0	10-15
	8.0		25.0	22.0	25-30
	9.0		12.0	10.0	38-48
18.0	7.0	0.14	55.0	42.0	10-15
	8.0		24.0	18.0	30-40
	9.0		22.0	10.0	32-42
19.0	7.0	0.16	28.0	22.0	28-35
	8.0		23.0	14.0	32-38
	9.0		16.0	10.0	35-45

This table shows that with the approximate 30 per cent reduction by cold rolling stated by the authors as being necessary to insure high elastic properties, there is a distinct advantage in the low nickel high carbon alloys in maintaining a high range of ductility.

The paper shows also the interesting observation that insofar as bending is concerned, there is little correlation between this property and the percentage elongation. This is because of the local rather than general elonga-

tion which occurs in the high nickel alloys. Such local elongation affects, of course, a lowering of the total percentage over a longer gage length. As has been pointed out, if a shorter gage length were used in the tensile test the percentage elongation in the steels of different analyses would be more nearly the same. In my opinion, however, the fact that a steel simply may bend without breaking is not a valid reason for its use in a highly stressed structure. Local elongation in these alloys seems to me to indicate that undesirable local constrictions may occur in the bend itself. It indicates, also, that the steel has been cold-rolled almost to its total capacity. It seems to me, therefore, that an appreciable amount of general plastic deformation (ductility) is as necessary in steels for highly stressed structures as high elastic properties. It has been my experience that where a compromise must be made between elastic properties and strength on the one hand, and ductility on the other, a slightly more ductile steel is preferable to a slightly "stronger" one.

I think this paper adds greatly to the rapidly increasing knowledge of the properties of these very useful steels.

Authors' Reply

It should be admitted that the friendly and constructive criticism is investigators' reward. That our attempt to correlate and gather together the fundamental information about the characteristics of one of the most important type of alloy steels was so kindly reviewed by men engaged in various branches of engineering is most gratifying. We will be even more pleased if our data pave the way for the use of stainless alloys in the installation, where such use was delayed because of the absence of data. We feel justified in expressing such hope when we hear such authority on airship construction as Mr. Paul Ffield tells us, in his discussion, that the presented data might have helped him when he was actively engaged in work on airships.

Mr. Ffield has had an enviable engineering experience and we can fully comprehend his disappointment when in testing chromium-nickel austenitic alloys he encountered proportional limits of as low as 5000 pounds per square inch on 200,000 pounds per square inch tensile strength material. The explanation for this, may we be permitted to call it "abnormal" finding, will, we believe, in the difficulties encountered be found experimental procedure. The characteristics of the colled-rolled materials are such, to quote the expression used in the manuscript, that "the yield point is ill defined and in no portion of the stress-strain curve may an elongation at constant load be found." It is our belief that the "elastic" properties of the alloys as above defined will not correctly portray the service behavior of the metal, especially if the metal is subjected to stresses greater than those that are in strict accordance with above given definition. For this reason we used the proof stress, a definite mechanical property, which despite its apparent artificiality, best satisfies in our opinion the engineering needs. Here is the reason for our belief, in studying "proof stress" of various alloys we were able to differentiate between such alloys and properly classify them, while similar classification on the basis of proportional limit would have produced discordant results.

We trust that Mr. Ffield shares our faith in proof stress. His remarks indicate that he studied this particular characteristic of the metals with con-

siderable attention. We are in complete agreement with his observation that the proof stress can be raised by means other than cold rolling. The method that he suggests, namely, preloading the material above the yield point, should result in higher proof stress. We believe this could have been predicted from other characteristic behavior of this type of alloys.

Another method would be that of age hardening, especially in those alloys in which age hardening characteristics are enhanced by the addition of small quantities of other elements. Work, now in progress, will soon determine the relative advantages of several methods of raising proof stress values.

Mr. Ffield is quite correct in his observation that certain factors besides those of composition and cold work influence the tensile properties. One of such factors, definitely established, is the rate of cold working or in other words the number of passes in which a certain amount of cold reduction is accomplished. One may except as much as 25,000 pounds per square inch difference in tensile strength by reducing the material 46 per cent in either one or five passes. About the same difference exists, so we found, when 75 per cent reduction was accomplished in 38 passes instead of 20. As a general rule it may be stated that the greater the number of passes the higher would be the tensile strength.

Regarding the direction of test specimen: The test specimens for mechanical properties were cut in the direction parallel to that of rolling. We find that physical properties, except bending, are affected relatively little by the location of the sample relative to direction of rolling. On the other hand the bend tests yield very different results depending on the direction in which the samples are taken. For this reason all bending tests were made in direction transverse to rolling since it is in this direction that the bending characteristics are the worst.

From the discussion sent to us by Mr. Olt and Mr. Burns, we surmise that we must have been insufficiently explicit while discussing our own results. The way we expressed the relation between composition and the available proof stress was as follows:

> "The rate of increase of proof stress induced by cold working is definitely slower in the more stable alloys than in the less stable alloys, as might have been anticipated."

As a matter of fact the original manuscript contained the curve so kindly brought to our attention by Mr. Olt and Mr. Burns. It was deleted later for reasons of desired brevity of presentation. At any rate, we fully realize that the analysis does have an influence, but this influence is not comparable with that of cold work. We are thankful to Messrs. Ffield, Olt and Burns, because they give us an opportunity to enlarge upon and clarify our own statement.

As to the slight discrepancies in numerical values for various mechanical properties, we should like to suggest that the figures for tensile strength are wholly within experimental error. Many variables are to be reckoned with in testing thin sheet samples. We preferred to give actually obtained values rather than average them up. The difference in actual values for elongation in very ductile alloys we of course noted. Our explanation is that in the majority of cases the samples break in such a way that the matching of two halves is a matter of personal equation.

Investigation of alloys of as low a chromium and nickel content as listed in Table A of Messrs. Olt and Burns' discussion has not been included in our program. We should like to suggest that these alloys are so "unstable" that the true evaluation of their properties is very difficult. We believe that it is upheld by the examination of the table and comparison of results. Samples 1 and H-8 are for all practical purposes of the same analysis except for carbon. In this case higher carbon produced lower $\frac{YS}{TS}$ value (last column, Table A). In alloys 3 and H-5, only slightly higher in chromium content (0.8-1.0 per cent), the action of carbon is reversed. The higher the carbon content the higher is the $\frac{YS}{TS}$ value.

The obtainable properties we believe to be influenced by "hardening while testing" and every factor (of which there are many) that alters the extent of the hardening will influence the final results more than difference in composition.

As to the relative corrodibility of these alloys our results, conducted on whole series of various compositions, would indicate the lowered resistance to corrosion. Such alloys certainly would not pass standard nitric acid test, nor would they yield totally satisfactory results, even under salt spray tests. This should not be interpreted as denying the usefulness of these alloys, only that they should be used with discrimination.

We have never failed to find most constructive criticism and suggestions in communications which are our privilege to receive from Mr. Franks. The questions concerning the impact values and the fatigue properties are and should be of much interest, especially the latter, and we are working at present on this very problem. The impact characteristics of fully softened austenitic alloys are so excellent that unless they are most profoundly and unexpectedly affected by the composition they still would be altogether satisfactory. The figures submitted by Mr. Franks on two different analysis steels uphold, in a way, our contention, yet frankly, we did not expect such a large difference in impact toughness.* Thus the investigation of toughness as a function of the composition and amount of cold reduction appears quite desirable after Mr. Franks' pertinent remarks.

Equally pertinent is the suggestion concerning the properties of cold-rolled materials at low temperatures. The preliminary work done some time ago on an alloy of 18-8 composition indicated that the influence of low temperatures is rather negligible. No attempt, as we recollect, was made to correlate properties at low temperatures with composition. Colbeck, MacGillioray and Manning in their recent publication inform us that liquid air temperature has no effect upon impact toughness. These researches, here and abroad, did not take into account the effect of temperature on cold-worked material. We agree with Mr. Franks that such data would be highly desirable and, in fact, we have this work already under way.

We were very much interested in Mr. Franks' thoughts in regard to elongation and reduction of area. Our own observation would not agree with his

*Incidentally, the values for yield point as given appear unexpectedly high.

belief that "when more stable steels fracture a greater reduction in area is obtained." We obtained in some of the unstable medium or high carbon alloys very high values for the reduction of area, altogether comparable with those obtainable in more stable alloys. As a permissible side remark I should like to suggest that some of us are wondering wherein lies the importance of determining the reduction of area? This very question was discussed not long ago with leading men in this field and the opinions are far from being unanimous, the "unbelievers" predominating. In austenitic alloys one thing appears to be quite definitely established; values for the reduction of area are very poor indication of other properties of the alloys. In our paper we refrained from calculating reduction of area because the measured areas were so small that even slight, unavoidable experimental error would have nullified or at least seriously hampered the evaluation of the data.

It is always gratifying when the results of an investigation are upheld by the work of other independent investigators. Mr. Winlock has been kind enough to make such a statement. The table which was summarized by Mr. Winlock, as well as the tables which we incorporated in our publication, seem to indicate that there is very little correlation between bending characteristics and the percentage of elongation. So far we are in perfect agreement with Mr. Winlock. We are inclined, however, to disagree with his subsequent deductions, to quote him, "Local elongations in these (that is high nickel alloys) alloys seems to me to indicate that undesirable local constrictions may occur in the bend itself. It indicates, also, that the steel has been cold-rolled almost to its total capacity." We find no justification for such belief in many tests that have been made. However, we are in full agreement with the very sensible deduction as proposed by Mr. Winlock, namely, that in highly stressed structures, it is as necessary to have appreciable amounts of ductility as it is to have high elastic properties. For that matter, this belief may be said to be one of the reasons why this extensive work has been undertaken. As Mr. Winlock is well aware we can produce at will, high elastic properties in stainless steels by what may be termed "excessive amount of cold work," but with subsequent limited ductility. In recounting in the paper the results of our investigation it was our aim to show that high elastic properties may be accompanied by fair ductility.

The written discussions to which we attempted to reply in the same friendly and impartial spirit in which they were written, also many pleasant oral discussions which we held with our colleagues, make us feel that the information contained in our publication was needed and timely. We shall continue our efforts in the same direction hoping for the same constructive and much appreciated criticism which has already been so freely given to us.

EFFECT OF OVERLOAD ON THE FATIGUE PROPERTIES OF SEVERAL STEELS AT VARIOUS LOW TEMPERATURES

By H. B. Wishart and S. W. Lyon

Abstract

Studies at several low temperatures were made by the authors of the effect of overload on the fatigue properties of an S.A.E. 1020 steel, a 0.75 per cent carbon steel, and a 3.0 per cent chromium steel. The probable damage diagram method of testing was used, because it provided the best organized means so far devised to evaluate the extent to which cycles of overstress above the endurance limit of a material damage that material.

In order to study the variation of damage at low temperatures, diagrams under controlled conditions were determined at +70, +10, —20, and —40 degrees Fahr. Specially constructed high speed fatigue testing machines were used to obtain the data for the construction of the damage diagrams.

Results of the tests indicate that there is little or no change of damage with change of temperature. This coupled with the fact that the endurance limits of the materials increased with decrease of temperatures suggests that for the metals tested, range of stress rather than temperature may be the critical factor in evaluating damage. The results of the tests also seem to indicate that the present method of damage determination is inadequate to provide a highly accurate method of damage evaluation and that changes in the method are desirable.

Introduction

CORRECTLY designed machine and structural members that are subjected to repeated stresses should not fail in service under normal operating conditions. That such fatigue failures are a rather common occurrence is evidence that during their life, these elements were subjected to cycles of definitely damaging overstress of a magnitude greater than the endurance limit of the materials. Little organized data is available relative to the damaging effect of cycles of over-

A paper presented before the Eighteenth Annual Convention of the Society held in Cleveland, October 19 to 23, 1936. The authors, H. B. Wishart and S. W. Lyon, are Special Research Assistants, University of Illinois, Urbana, Illinois. Manuscript received June 22, 1936.

stress either as to magnitude or repetitions of such stresses that will ultimately cause failure[1]. The data presented in this paper were obtained with the purpose of contributing some additional information on the general problem of the effect of overload on the fatigue properties of materials.

The second phase of this paper deals with the effect of subnormal temperatures upon "damage" as found for the materials tested. It is the general impression that there are marked changes of the properties of metals with lowering of temperature. As opportunity was recently afforded the writers to make a series of tests at low temperatures, using one of the cold rooms at Wright Field, Dayton, Ohio, these data were obtained along with other test results and are presented as offering some information on the variation of the "damageability" of three steels when subjected to temperatures as low as —40 degrees Fahr.

Methods of Testing

To study the problem of the effect of overload on the fatigue properties of the three steels tested, the "probable damage diagram" method presented by French[2] was used. It presents the only organized means so far devised to study this problem.

This method provides for the establishment of the endurance limit or regular S-N diagram. The regular S-N diagram is the right hand curve, see Fig. 3. The construction of the left hand curve or "damage curve" which completes the boundry of the "damage range" is the next consideration. This curve is determined by overstressing a specimen at a stress above the endurance limit for a certain number of cycles. The specimen is then run at the endurance limit stress. If the specimen fails when run at the endurance limit the metal is assumed to have been damaged—if the specimen did not fail, then no apparent damage was done. This process was repeated, keeping the same overstress but varying the number of cycles of overstress until at least two specimens were tested that did not break and two that did break were secured, see Fig. 3—solid circles are specimens that did not break, hollow circles are specimens that did break. This method then determines the cycles at which damage for this particular overstress starts. The same method is now used but at a different

[1]H. W. Russell and W. A. Welcker, Jr., "Damage and Overstress in the Fatigue of Ferrous Metals," Preprint, A.S.T.M., 1936.

[2]H. J. French, "Fatigue and the Hardening of Steels," Transactions, American Society for Steel Treating, Vol. 21, October, 1933, p. 899.

overstress to locate other damage points—a curve drawn through these points is the "damage curve." Metal subjected to any combination of stress and cycles which falls within the area between the "damage curve" and regular S-N diagram, "Damage Range" is considered as damaged.

Tests and Test Equipment

Three materials, an S.A.E. 1020 steel, a 0.75 per cent carbon steel, and a 3.0 per cent chromium steel were used for test. The S.A.E. 1020 specimens were machined from $\frac{5}{16}$-inch diameter bar stock while the specimens for the other two materials were cut from rail heads.

Four series of specimens for each material were prepared and were tested at four temperatures, +70, +10, −20, and −40 degrees Fahr.

The chemical composition, heat treatment, and physical properties are given respectively in Tables I, II, and III.

Table I
Chemical Composition of Steels Tested

Material	Per Cent C	Mn	P	S	Si	Cr
S.A.E. 1020 Steel*	0.20	0.45	0.045	0.055	—	—
0.75 per cent Carbon Steel	0.75	0.84	0.030	0.023	0.15	—
3.0 per cent Chromium Steel	0.25	0.69	0.013	0.020	0.25	2.89

*Approximate Chemical Analysis

Table II
Heat Treatment of Steels Tested

Material	Treatment
S.A.E. 1020 Steel	1550 degrees Fahr. for ½ hr. Cooled in Air
0.75 per cent Carbon Steel	Tested as Received. (Hot-Rolled)
3.0 per cent Chromium Steel	Tested as Received. (Hot-Rolled)

Table III
Physical Properties of Steels Tested

Material	Per Cent Elong. in 2 in.	Yield Strength 0.2 Per Cent Gage Length	Tensile Strength	Endurance Limit			
		lb./sq. in. +70 degrees Fahr.		lb./sq. in. +70°F.	+10°F.	−20°F.	−40°F.
S.A.E. 1020 Steel*	25	50,400	62,700	31,000	41,000	45,000	48,000
0.75 per cent Carbon Steel**	12	59,600	130,400	63,000	67,000	67,000	69,000
3.0 per cent Chromium Steel**	16	103,100	185,200	94,000	94,000	97,000	100,000

*Tension Specimen 0.150 inches in diameter
**Tension Specimen 0.505 inches in diameter

Fig. 1 is a photograph of one of the special high speed fatigue testing machines used for the tests. As can be seen, this is of the cantilever type, and is loaded by means of a weight sliding on a calibrated beam that reads directly in inch-pounds bending moment. The speed of the machine is variable between 8000 and 14,000 revolutions per minute; a speed of 9000 revolutions per minute was selected

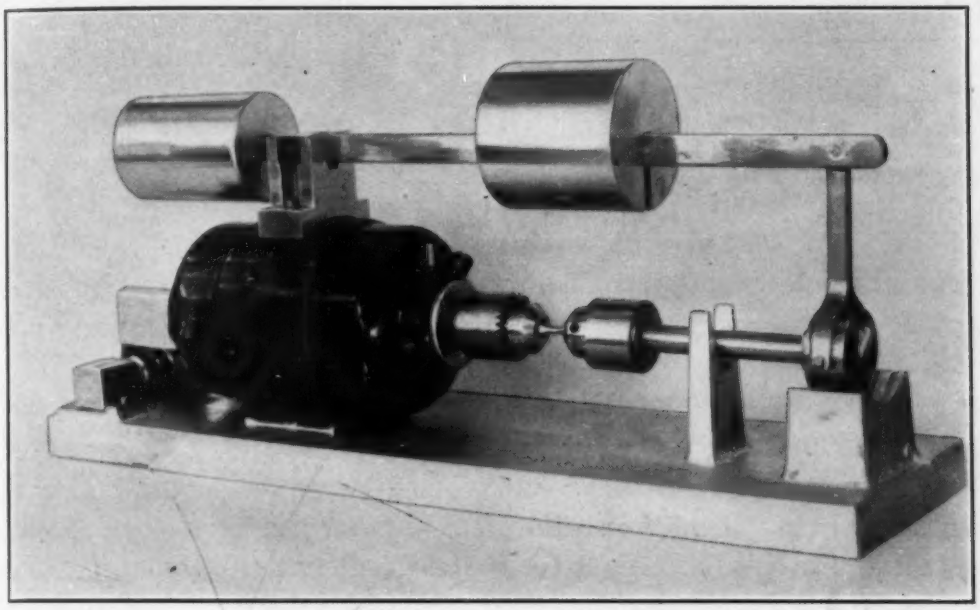

Fig. 1—High Speed Fatigue Testing Machine.

for the tests herein reported. The overall dimensions of the machine are length 17 inches, width 5 inches, and height 7 inches. For the duration of the tests an auxiliary fan was attached to the motor shaft between the motor housing and specimen chuck in order to assure as small a temperature gradient as possible between the surrounding air and motor during tests. Temperature readings taken at intervals by a thermometer placed in a well on the motor housing gave an 8-degree gradient between motor temperature and atmosphere. Measurement of specimen temperatures during operation offered considerable difficulty and various methods tried indicated that the gradient between specimen and the testing room atmosphere was about 4 degrees Fahr.

Lubrication of the high speed machines, especially at the low temperatures, proved to be an important consideration. While not entirely satisfactory, an S.A.E. 20 oil cut with kerosene to assure requisite viscosity at the operating temperature gave the best results of the many blends tried. An excess of oil proved as troublesome as

did a deficiency, for both conditions promoted undue friction and ultimate bearing failure.

Fig. 2 shows the type of fatigue specimen used. In order to assure uniformity of finish and freedom from annular scratches a uniform procedure of polishing was used. This consisted of treat-

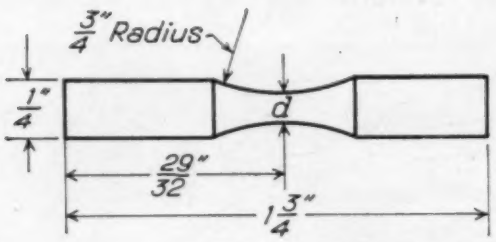

Fig. 2—High Speed Fatigue Test Specimen.

ment with four grades of polishing paper so applied that final polishing marks were all in a longitudinal direction, thus minimizing any possible notch effect.

The cold room at Wright Field in which the tests at low temperatures were conducted consisted essentially of a room 10 feet square by 8 feet high, insulated with 12 inches of cork. The refrigeration equipment consisted of a 12-ton capacity carbon dioxide installation which served to maintain temperatures down to −50 degrees Fahr., with a temperature control of ±5 degrees Fahr.

Results of Tests

Figs. 3, 4, and 5 give the damage diagrams at the four testing temperatures for the three steels tested. It may be noted that with the exception of the S.A.E. 1020 steel there is no marked change of damage range with change of temperature. For the S.A.E. 1020 steel, however, there is a marked progressive narrowing or reduction of the diagrams with lowering of temperature. While there are insufficient data to justify any definite conclusion, the fact that this steel is the only one of the three which has marked ductility suggests the possibility that ductility may have a controlling influence on the damaging effect of cycles of overstress on steel. It also might be well to remember in this connection, that the ductility as measured by the static tension test decreases with decrease of temperature[3] and such decrease could be expected to affect the character of the damage diagrams.

[3]H. F. Moore, "Progress Report of the Joint Investigation of Fissures in Railroad Rails," Engineering Experiment Station, University of Illinois, Urbana, Ill., Preprint No. 4, p. 19.

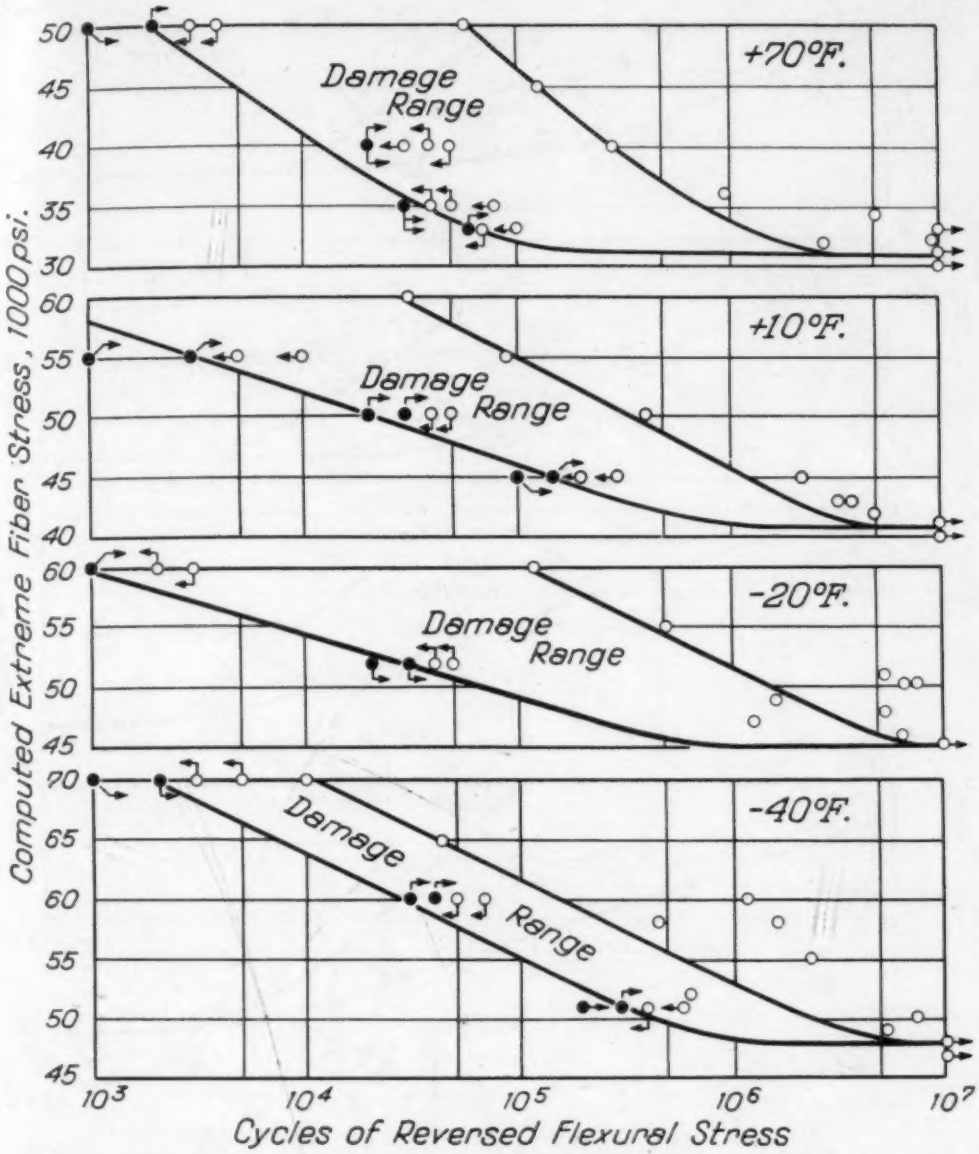

Fig. 3—Damage Diagrams for an S.A.E. 1020 Steel at +70, +10, −20, and −40 Degrees Fahr. (Solid Circles with Arrows Are Specimens not Damaged by Overstress—Hollow Circles with Arrows Are Specimens Damaged by Overstress.)

As mentioned in the previous paragraph, the damage diagrams for the 0.75 per cent carbon steel and 3.0 per cent chromium steel changed little in character with change of testing temperature. While the relative areas of the diagrams varied but little, there was a definite increase of the basic endurance limit for these two materials with decrease of temperature, as shown in the diagrams, Figs. 4 and 5. It is suggested, then, from these test results that, for these materials, the range of stress or the amount of overstress above the endurance limit determines the character or area of the damage range rather than change of temperature. Such being the case, further tests are

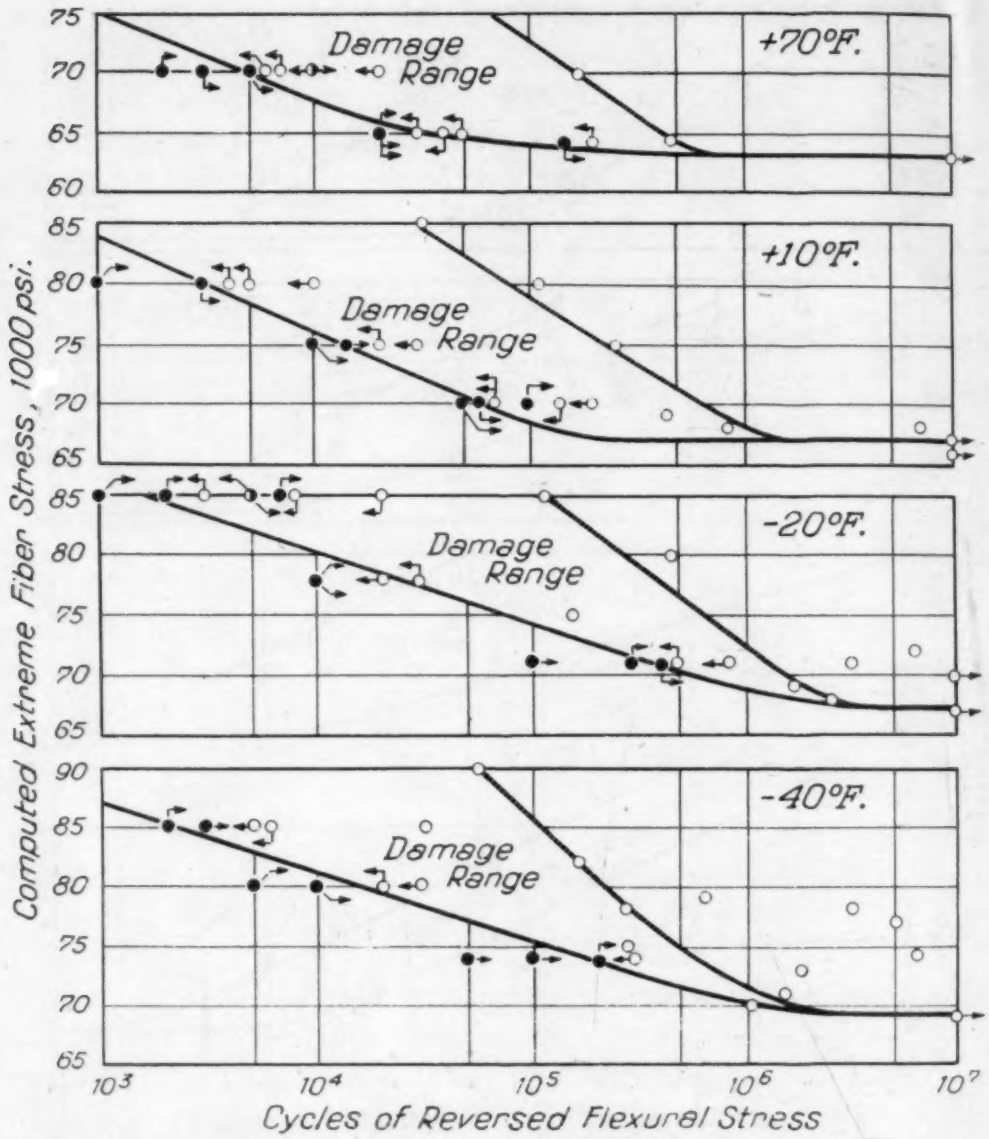

Fig. 4—Damage Diagrams for a 0.75 Per Cent Carbon Steel at +70, +10, −20, and −40 Degrees Fahr. (Solid Circles with Arrows Are Specimens not Damaged by Overstress —Hollow Circles with Arrows Are Specimens Damaged by Overstress.)

indicated as being desirable to determine whether this conclusion may hold for other materials possessing slight ductility.

During the performance of these tests considerable scatter was encountered. It was determined that the major portion of this scatter was traceable to changing endurance limit. Nonuniformity of the as-received material with resulting variable properties accounts for the changing endurance limits. Any shifting of the endurance limit, whether above or below the basic value upon which the diagram was based, will, of course, result either in premature failure or no failure at all which will in any event vitiate the results. A

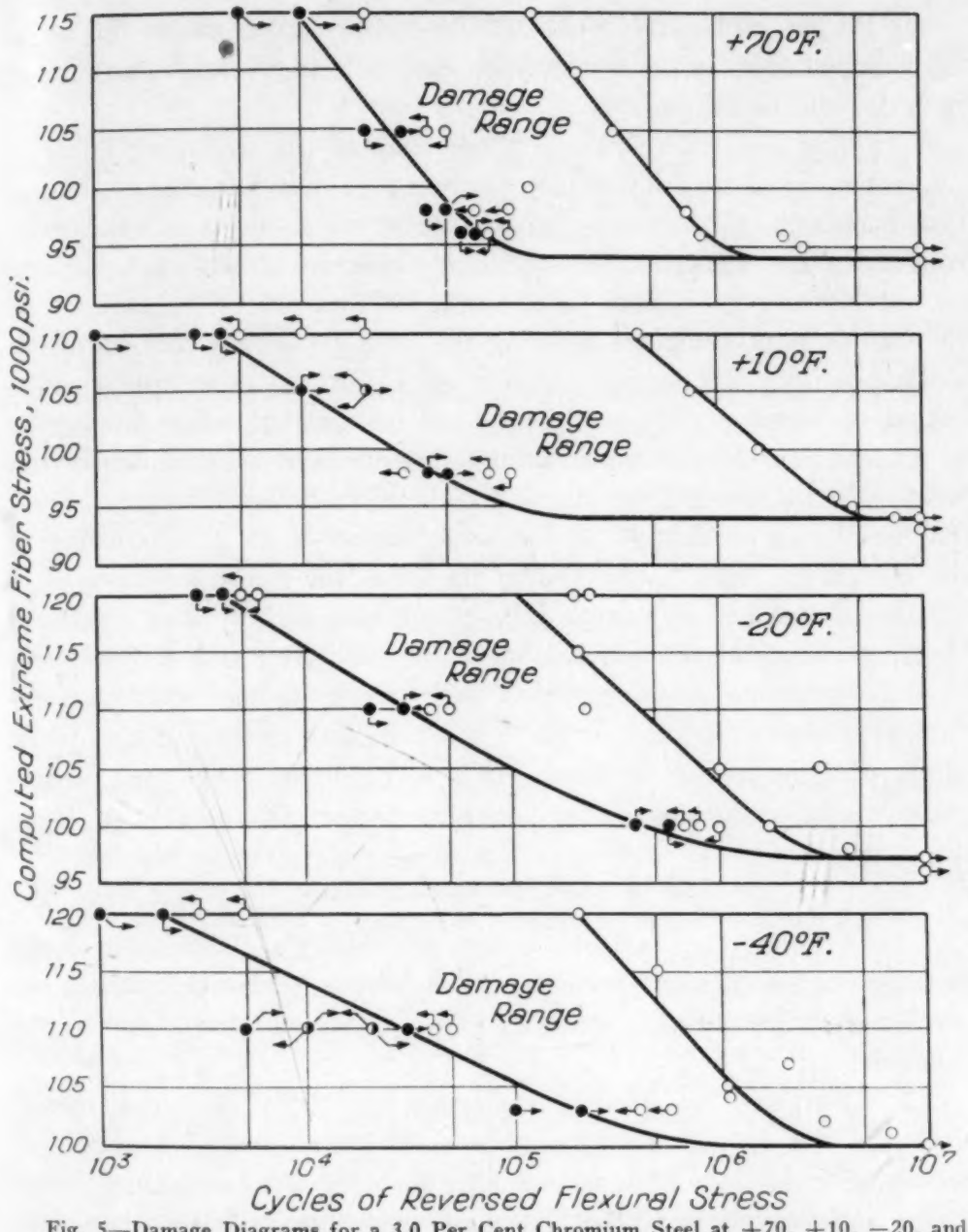

Fig. 5—Damage Diagrams for a 3.0 Per Cent Chromium Steel at +70, +10, −20, and −40 Degrees Fahr. (Solid Circles with Arrows Are Specimens not Damaged by Overstress —Hollow Circles with Arrows Are Specimens Damaged by Overstress.)

possible improvement in the method has been suggested by Moore[4]. The method as now used provides that the overstressed specimens used in constructing the damage line be re-run at the endurance limit. To obviate the objection introduced by possible change of endurance limit due to nonuniform material, it is proposed to re-run the overstressed specimens at some percentage of the endurance limit stress

[4]H. F. Moore, Research Professor of Engineering Materials, University of Illinois, Urbana, Illinois.

—say 90 per cent—and construct the damage diagram on this basis. It is hoped that by so doing false results due to nonuniformity of material will be minimized.

In studying the results of these tests of the damaging effect of overstressing of steel it became apparent that only one side of the picture had been considered—perhaps the worst side—as far as service conditions are concerned. In a machine element in service, occasional periods of overstress may be encountered that are of a duration less than an amount sufficient to bring the total cycles for that particular instance within the damage range area. Work of other investigators —that of Kommers[5] in particular—has shown that succeeding cycles of stress below the endurance limit following limited amounts of overstress will for some materials at least tend to have a "healing effect" that results in no change or for some materials an actual raising of the endurance limit. Such being the case, the questions arise as to whether "damage" is cumulative or not and within what limits the "healing effect" above noted is operative. The need of further study of understressing in conjunction with overstressing is strongly indicated if not imperative to furnish a complete picture of this phase of the fatigue testing problem. It is to be hoped that other investigators may find the time and opportunity to further study the problem.

Summary of Conclusions

1. Of the three steels tested the S.A.E. 1020 showed the greatest change of damage range with variation of temperature. The greater ductility of this material is a possible explanation of this behavior.

2. For the less ductile materials, the 0.75 per cent carbon steel and the 3.0 per cent chromium steel, the range of overstress above the endurance limit is indicated as being the controlling factor in the determination of the "damage range" rather than change of temperature.

3. The endurance limits of the steels tested increased with decreasing temperatures.

4. The results of the tests indicate that the present method of damage determination is inadequate to provide a sufficiently accurate method of damage evaluation and that changes in the method are desirable.

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[5]J. B. Kommers, "Understressing and Overstressing on Iron and Steel," *Engineering News Record*, Vol. 114, April 18, 1935, p. 550.

DISCUSSION

Written Discussion: By J. B. Kommers, University of Wisconsin, Madison, Wis.

During the past several years a number of investigators of fatigue phenomena have become interested in the effect of periods of overstress on the fatigue strength of metals. Most of the investigators have employed the same method used by Wishart and Lyon in determining a probable damage curve.

In the experiments which the writer has carried on he has employed a somewhat different method. After the periods of overstress, instead of subjecting the specimen to a stress at the virgin endurance limit, the writer has determined the new endurance limit, and he has taken as a measure of damage due to overstress the percentage by which the overstress has reduced the virgin endurance limit. Tests have been carried out on a 0.27, a 0.50, a 0.62 per cent carbon steel, and on a gray cast iron with an ultimate tensile strength of 33,600 pounds per square inch. For these four materials the results show that overstress causes damage in all cases except when the percentage of overstress is quite small and the number of cycles of overstress is small compared with the number of cycles which would cause failure at that stress.

It is evident that these results are different from the results obtained by the authors, because their damage curves show that the three materials tested could be subjected to fairly large numbers of cycles without damage.

In tests reported by Russell and Welcker at the A.S.T.M. convention in June, 1936, three of their materials showed a horizontal damage curve, and one other was practically horizontal. Such curves indicate that damage was done even at small percentages of overstress and at small numbers of cycles.

For the present, therefore, the conclusion must be drawn that some materials are much more susceptible to damage by overstress than others. It will be of interest to attempt to determine whether resistance to overstress can be correlated with any of the ordinary static properties of metals.

One of the interesting facts brought out by the experiments of Wishart and Lyon is that the endurance limit is increased with decrease of temperature. This fact raises the question as to whether there is developed a fairly intense local heat at microscopic areas due to repeated stresses, which may have a damaging effect on the metal. Such local high temperatures, if they exist, would tend to be reduced when the specimen is very cold.

Since many machine elements fail because of the presence of notches or other abrupt changes of cross section, there is considerable practical importance in determining the resistance to overstress of notched specimens. The writer has carried on such tests, and Russell and Welcker have reported tests on square notched specimens in their A.S.T.M. paper. From the latter results it is clear that the damage curves for standard and notched specimens of the same material need not be similar. In quite a number of cases the damage curve for notched specimens was closer to the original endurance curve.

Oral Discussion

A. V. DE FOREST:[1] It is gratifying to find that there is not as much indication of detriment due to low temperature in the fatigue as in the impact test.

[1]Associate professor, mechanical engineering, Massachusetts Institute of Technology.

Everybody thinks of low temperatures as being detrimental, damaging and dangerous, and yet, here are, as far as I know, the first real results that indicate that low temperatures, as far as fatigue is concerned, are not to be greatly feared. Of course, that has nothing to do with the impact problem which is a different kind of mechanism.

It is not particularly indicated by anything that I have read as to what the position of the damage line really indicates—desirable or undesirable. The damage line occurs somewhere, and the gap between the damage line and the fatigue curve probably represents the duration of the time during which the crack, once formed, is growing. Now, we rather imagine that it is desirable to have cracks grow slowly, because then there is a better chance of picking out the crack before it has actually come to a catastrophe. In a great many cases, parts are examined at intervals and cracks are found in such a proportion compared with the number of broken parts, that it is quite evident that we are finding a good deal of damage in the form of the crack before the crack has gone entirely through, so it would seem desirable to have a damage line that started early, and a slow growing rate of crack. If we are going to have the same fatigue limit in any case we might just as well have the crack there as an indicator, as a safety precaution, rather than a type of steel in which the crack does not start until very late in the life of the part and then goes through the part extremely rapidly.

There is some hope of finding out which steels do what, but it will take a great deal more of this damage line work before we are really confident as to what particular safety factor exists in the parts as used. This is an excellent paper with which to start that line of thought.

Authors' Reply

The suggestion made by Kommers as a possible explanation for the change of endurance limit with temperature change is reasonable. Other theories have also been advanced but so far none seem to entirely explain the effect of low temperatures on metals. The main difficulty in explaining the low temperature effect on metals is that all metals are not susceptible, or at least to an appreciable extent, to low temperature embrittlement. Most of the theories explain the action of a metal when it is embrittled by low temperatures but they cannot be applied as well when explaining why a metal was not embrittled or appreciably embrittled in another case. The authors are very interested in this problem and would like to know just what happens in some metals to cause a change in the physical properties with temperature change while in other metals there is no appreciable change.

In regard to whether the resistance to overstress can be correlated to the ordinary static properties a small amount of work of this nature was undertaken by the authors. It was found that the static properties could not be readily correlated with the resistance of overstress. Far too few tests were made, however, to make any definite statements or to draw any conclusions.

The question of whether it is desirable to have a wide or small damage range as brought up by Mr. de Forest is a very debatable one. Much consideration has been given this question by the authors and so far no definite decision

has been reached by them as to which condition might be best. The study so far has been made by subjecting the metal to initial periods of overstress followed by determining the damage or weakening of the material due to this overstressing. Thus, a metal with a wide damage range would receive more damage for a given amount of overstress and have its endurance limit lowered to a much greater extent than a metal with a narrow damage range. The study of the damage due to overstressing as presented in this paper is perhaps the most severe case that could occur. In order to get a complete picture of the mechanism of "damage" done to a metal the effect of understressing must also be brought into the picture. In other words, most parts are designed to be used at some stress below the endurance limit. When occasional overstress occurs, is the damage done by this occasional overstress healed by the numerous cycles of understress or what does happen? In order to answer Mr. de Forest's question then, all of these conditions must be considered and so far the data for doing so are not available.

The data presented in this paper are part of a second series of low temperature tests made by the authors. The second test was made for the purpose of studying the effect of low temperatures on the "damage" done by overstressing of a metal. The first series of tests[3] were made to determine the effect of low temperatures on the physical properties of metal. In this test it was realized that the important property to consider as far as low temperatures were concerned was the ability of a metal to withstand impact. The variation in the endurance limit, tensile strength, and hardness would cause little concern.

[3]*Metal Progress* November 1935, p. 46.

SLIP, TWINNING AND CLEAVAGE IN IRON AND SILICON FERRITE

By C. S. Barrett, G. Ansel, and R. F. Mehl

Abstract

As a basis for a better understanding of the effect of alloy additions upon the physical properties of iron and of annealed steels, the elementary mechanical and crystallographic processes of slip, twinning and cleavage are studied, this paper dealing with these phenomena in iron-silicon alloys.

Regarding the crystallographic features, slip in iron is found to be on {1 1 0}, {1 1 2} and {1 2 3} planes at all temperatures investigated, but in silicon ferrite it is discovered that low deformation temperatures or high silicon contents cause slip to be confined wholly to {1 1 0} planes. Twinning and cleavage planes are found as previously reported, {1 1 2} and {1 0 0} respectively.

Schematic curves are drawn to show the relative variation of the stresses necessary to produce slip, twinning and cleavage; and the mechanical properties, particularly Pilling's curve of brittleness, are interpreted in terms of these curves

THIS research, together with related ones now being conducted, is intended to furnish a basis for a better understanding of the effect of alloy additions upon the physical properties of iron and of annealed steels. The elementary mechanical and crystallographic processes of slip, twinning and cleavage are considered as the basis of this understanding, and in this paper these processes are analyzed in iron and iron-silicon alloys and are correlated with the well known variation of brittleness in the materials as a function of temperature and silicon content. A new phenomenon has been discovered in the course of the work; namely, that the addition of an element in solid solution may change the mechanism of slip, in this case by inhibiting

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A paper presented before the Eighteenth Annual Convention of the Society held in Cleveland, October 19 to 23, 1936. Of the authors, who are members of the Society, C. S. Barrett is associated with the Metals Research Laboratory and with the Department of Metallurgy of the Carnegie Institute of Technology. G. Ansel is an assistant in the Metals Research Laboratory. R. F. Mehl is director of the Metals Research Laboratory and head of the Department of Metallurgy, Carnegie Institute of Technology, Pittsburgh. Manuscript received June 25, 1936.

slip on certain crystallographic planes. These experiments have also added an example to the small list of materials whose slip mechanism is known to be altered by temperature. In the first portion of this report the crystallographic features of slip, twinning and cleavage are treated—including the controversial subject of the nature of slip in iron. The second part of the paper deals with the critical stresses required for these processes and presents curves showing the relative variation of these properties with temperature and composition, and schematic diagrams which afford a fundamental point of view for understanding the characteristic mechanical properties of silicon ferrite.

A. The Crystallographic Features of the Processes

The crystallographic nature of plastic deformation in metals possessing body-centered lattices, particularly alpha iron, is still a matter of some uncertainty in spite of lengthy researches over a long period of years. With crystals of β brass Göler and Sachs[1] and also Elam[2] have observed slip lines that correspond to slip on the planes of greatest atomic density, {1 1 0}, and in the direction of the closest packed strings of atoms, [1 1 1]. Yet Taylor[3] found that no single crystallographic plane of slip would adequately explain the change of shape of β brass crystals when pulled in tension, although the data required [1 1 1] as a slip direction. Elam,[4] in the most recent study of this material, found that the slip lines first appearing, while usually traces of {1 1 0} planes, were occasionally traces of {1 1 2} planes, but that as deformation progressed they ceased to correspond to any definite crystal planes; the change of shape in tension and the change in crystal orientation, as followed by X-ray photograms also confirmed this result and led to the further conclusion that [1 1 1] is not always the slip direction. Goucher[5] concluded, on the basis of an X-ray and microscopic study, that tungsten slips on {1 1 2} planes in a [1 1 1] direction. While data on these body-centered crystals are quite discordant, the data on iron are still more so. Many suggestions as to the crystallographic mechanism of slip in iron need not be discussed here—particularly the earlier ones—as they were based

[1] Göler and Sachs, *Naturwissenschaften*, Vol. 22, 1928, p. 412.
[2] C. F. Elam, *Nature*, Vol. 133, 1934, p. 723.
[3] G. I. Taylor, *Proceedings*, Royal Society, London, Vol. A118, 1928, p. 1.
[4] C. F. Elam, *Proceedings*, Royal Society, London, Vol. A153, 1936, p. 273.
[5] F. S. Goucher, *Philosophical Magazine*, Vol. 48, 1924, p. 229, 800.

on data too fragmentary to be of much value now.[6] O'Neill[7] and Pfeil[8] concluded that in 1.8 per cent silicon ferrite and in purified iron, respectively, slip occurred on {1 1 2} planes in [1 1 1] directions. This conclusion, however, is not rigorous since it is based on observations of a limited number of slip lines and since slip on {1 2 3} planes was not disproved by the analysis.

Taylor and Elam[9] concluded from measurements of the distortion of iron crystals in tension and compression that slip was not confined to planes of a single form since the orientation of the slip plane appeared to lie at various positions between {1 1 2} and {1 1 0} planes. It was suggested that the deformation of iron could be compared with the deformation that occurs in a bundle of pencils when these slide over each other in groups and layers. A section along the slip direction [1 1 1], i.e., along the pencil axes, would show straight slip lines, but a transverse section would show irregular, uneven slip lines. Under these assumptions, the surface on which displacement occurs would be non-planar, or "banal," as designated by Taylor and Elam.

Fahrenhorst and Schmidt[10] objected that distortion measurements of the type employed by Taylor and Elam could not yield a unique solution to the problem if slip had occurred on more than one crystallographic plane, and that Taylor and Elam's data could equally well be explained by slip on individual planes and pairs of planes of the form {1 2 3}. As evidence that the slip plane is {1 2 3} and that the slip direction is [1 1 1] it was shown that only this mechanism, and not {1 1 0}, {1 1 2}, or "banal," could be consistent with the dependence of the following properties on crystal orientation: (1) the change in dimension of the cross section, (2) the change in orientation with elongation, and (3) the yield point. But this study is, in turn, open to objection. The three properties were not in satis-

[6] J. A. Ewing and W. Rosenhain, *Philosophical Transactions*, Royal Society, London, Vol. 67, 1900, p. 112.
F. Osmond and G. Cartaud, *Journal*, Iron and Steel Institute, Vol. 71, 1906, p. 444.
H. M. Howe, Metallography of Steel and Cast Iron, 1916.
F. C. Thompson and W. E. W. Millington, *Journal*, Iron and Steel Institute, Vol. 119, 1924, p. 67.
W. Rosenhain and J. McMinn, *Proceedings*, Royal Society, London, Vol. A108, 1925, p. 231.
M. Polanyi, *Zeitschrift für Physik*, Vol. 17, 1923, p. 42.
K. Weissenberg, *Zeitschrift für Kristallographie*, Vol. 61, 1925.
H. Mark, *Zeitschrift für Kristallographie*, Vol. 61, 1925.
G. Tammann and Müller, *Zeitschrift für Metallkunde*, Vol. 27, 1935, p. 187.

[7] Hugh O'Neill, *Journal*, Iron and Steel Institute, Vol. 113, 1926, p. 418.

[8] L. B. Pfeil, Iron and Steel Institute, *Carnegie Scholarship Memoirs*, Vol. 15, 1926, p. 319.

[9] G. I. Taylor and C. F. Elam, *Proceedings*, Royal Society, London, Vol. A112, 1926, p. 337.

[10] W. Fahrenhorst and E. Schmidt, *Zeitschrift für Physik*, Vol. 78, 1932, p. 383.

factory agreement with any of the theories; inhomogeneities in structure or other causes gave rise to variations in these properties of the same order of magnitude as the differences necessary to distinguish between the various theories. In fact, as Elam[11] pointed out, some of the crystals rotated as if the direction of slip is not always the [1 1 1] direction.

Some experiments recently made by Elam included, in addition to the analysis of distortion of the iron crystals, in the manner of the earlier work of Taylor and Elam, some measurements on slip lines formed in tension, together with X-ray measurements of the changes in lattice orientation, with results contradictory to the earlier ones: the initial slip appeared to be on {1 1 0}planes, subsequent slip did not, and the slip direction was not always [1 1 1].

The most rigorous determination of the slip plane in alpha iron that has been published is Gough's,[12] based on slip lines and calculations of stresses in a single crystal of decarburized mild steel subjected to alternating torsion. Excellent agreement was obtained with the assumption that slip occurred on {1 2 3} and {1 1 0} when the plane of maximum shear stress lay between these two, and on {1 2 3} and {1 1 2} when the plane of maximum shear stress fell between these, slip on each of the pairs occurring in the [1 1 1] direction common to both planes of the pair. In special orientations of the plane of maximum shear, slip occurred on planes of a single type, either {1 1 0}, {1 1 2}, or {1 2 3}. Sauerwald and Sossinka[13] disagreed with Gough and concluded that slip was confined to {1 2 3} planes at room temperature and to {1 1 2} planes at low temperatures, but the evidence presented in support of this theory was somewhat meager; it depended on measurements of portions of irregular slip lines on compressed blocks and the proper pairing (by trial and error) of the measurements on one side of a crystal with the measurements on another side. It may further be objected that at low temperatures mechanical twinning ("Neumann bands") would be encountered which must be carefully differentiated from slip lines. This, apparently, has not been done, although methods suitable for this have long been known.[14] Sauerwald and Sossinka's Fig. 2 shows markings, interpreted as slip lines, that have every appearance of being twins. Thus, the conclusion that the low temperature strain-

[11]C. F. Elam, *Proceedings*, Royal Society, London, Vol. A153, 1936, p. 273.
[12]H. J. Gough, *Proceedings*, Royal Society, London, Vol. A118, 1928, p. 498.
[13]F. Sauerwald and H. G. Sossinka, *Zeitschrift für Physik*, Vol. 82, 1933, p. 634.
[14]F. Osmond and G. Cartaud, *Journal*, Iron and Steel Institute, Vol. 71, 1906, p. 444.

markings are traces of {1 1 2} planes may be interpreted as a confirmation of {1 1 2} twinning rather than a proof of {1 1 2} slip.

Akulov and Raewsky[15] attempted to study slip lines in iron by depositing magnetic powder on the surfaces of deformed crystals, but without much success, for again the processes of slip and twinning were not differentiated, and only one of the various planes capable of producing the traces was considered in the analysis of traces.

It appears that with the single exception of Gough's alternating torsion experiment, all the attempts to determine the slip mechanism in alpha iron are vulnerable to criticism in one or more particulars: those based on slip lines are lacking either in the amount of data reported, in a rigorous analysis of the data, or in the differentiation of slip lines from Neumann bands; those based on measurements of the macroscopic distortion, on rotation of the crystal lattice, or on the dependence of the yield point on crystal orientation are inadequate to treat cases where slip occurs on more than one set of planes (and this is apparently a very common occurrence). Gough's work was confined to one composition (decarburized mild steel), one temperature (room temperature), and one method of loading (repeated torsion).

A change in the slip mechanism at elevated temperatures has been observed in aluminum,[16] magnesium,[17] and β-tin,[18] but there has been no adequate search for a temperature effect with alpha iron, nor for a change of the mechanism with composition in alloyed ferrites.

The work reported herein includes observations of the plane of slip in crystals of purified alpha iron, and of silicon ferrite of a range of compositions, with deformation over a range of temperatures. A method of analysis is used that takes into account all likely planes capable of producing the slip lines and that discloses whether or not any given set of slip lines may be explained by slip on a single plane. Methods of deformation were used that produced a sufficient number of reasonably straight and accurately measurable slip lines, and no measurements were made on curved or wavy lines. X-rays were used to determine the orientation of the crystals to an accuracy greater than that possible in the measurement of the slip lines, and

[15] N. Akulov and S. Raewsky, *Annalen der Physik*, Vol. 20, 1934, p. 113.

[16] Karnop and Sachs, *Zeitschrift für Physik*, Vol. 41, 1927, p. 116; Vol. 42, 1927, p. 283. Boas and Schmidt, *Zeitschrift für Physik*, Vol. 71, 1931, p. 703.

[17] E. Schmid, *Zeitschrift für Elektrochemie*, Vol. 37, 1931, p. 447.

[18] H. Mark and M. Polanyi, *Zeitschrift für Physik*, Vol. 18, 1923, p. 75. I. Obinata and E. Schmid, *Zeitschrift für Physik*, Vol. 82, 1933, p. 224.

in nearly all cases the X-ray work followed the deforming process, thus eliminating any errors due to reorientation in the deforming process. Care was taken to differentiate slip lines from mechanical twins and small cleavage cracks. All markings resulting from twinning and cleavage were analyzed by the same methods that were used for the slip lines. Thus not only were the planes of twinning and cleavage determined in the crystals of various silicon contents, but additional assurance was received that the X-ray photograms employed in analyzing slip lines were correctly interpreted.

With regard to previous work on the crystallography of twinning, it should be stated that Neumann bands in ferrite forming on {1 1 2} planes[19] have been shown by microscopic methods[20] and by X-ray reflections[21] to be deformation twins. The crystallographic plane delineated by these twins is the same in alloyed ferrite as in iron[22] and is unchanged by temperature.[23] Cleavage in ferrite is along cube planes[24] although in isolated instances cleavage on {1 1 0} or {1 1 2} planes also has been suspected.[25]

Materials and Treatment—The purified iron single crystals used in this work were prepared by Dr. M. Gensamer at the Carnegie Institute of Technology. The material before purification had a ladle analysis of 0.13 per cent carbon, 0.36 per cent manganese, 0.010 per cent phosphorus, 0.025 per cent sulphur and 0.09 per cent silicon. After being annealed for 2 days in nitrogen at 980 degrees Cent. (1795 degrees Fahr.), it was purified in wet hydrogen at 750 degrees Cent. (1380 degrees Fahr.) for 14 days. Single crystals were then

[19]Linck, *Zeitschrift für Kristallographie*, Vol. 20, 1892, p. 209.
O. Mügge, *Neues Jahrbuch Mineral*, Vol. 2, 1899, p. 55.
F. Osmond and G. Cartaud, *Journal*, Iron and Steel Institute, Vol. 71, 1906, p. 444.

[20]K. Harnecker and E. Rassow, *Zeitschrift für Metallkunde*, Vol. 16, 1924, p. 312.
H. O'Neill, *Journal*, Iron and Steel Institute, Vol. 113, 1926, p. 417.
L. B. Pfeil, Iron and Steel Institute, *Carnegie Scholarship Memoirs*, Vol. 15, 1926, p. 319.

[21]C. H. Mathewson and G. H. Edmunds, American Institute of Mining and Metallurgical Engineers, Technical Publication 139, 1928.

[22]C. H. Mathewson and G. H. Edmunds, preceding reference (3.78 per cent Silicon).
H. M. Howe, "Metallography of Steel and Cast Iron," 1916, (4 per cent Silicon).
Robin, *Revue de Metallurgie, Memoirs*, Vol. 8, 1911, p. 436, (1 per cent Phosphorus).

[23]F. Osmond and G. Cartaud, *Journal*, Iron and Steel Institute, Vol. 71, 1906, p. 444.
Sir R. A. Hadfield, *Journal*, Iron and Steel Institute, No. I, 1905, p. 248.
H. M. Howe, loc. cit.
L. B. Pfeil, Iron and Steel Institute, *Carnegie Scholarship Memoirs*, Vol. 15, 1926, p. 319.

[24]Sadebeck, Poggendorf, *Annallen der Physik*, Vol. 156, 1875, p. 554.
J. E. Stead, *Journal*, Iron and Steel Institute, 1898, p. 148.
Osmond, Frémond and Cartaud, *Revue de Métallurgie*, Vol. 1, 1904, p. 11.
Osmond and Frémond, *Comptes Rendus*, Vol. 141, 1905.
Hugh O'Neill, *Journal*, Iron and Steel Institute, Vol. 113, 1926, p. 417.
W. Fahrenhorst and E. Schmid, *Zeitschrift für Physik*, Vol. 78, 1932, p. 383.
F. Bitter, *Proceedings*, Royal Society of London, Vol. A145, 1934, p. 668.

[25]Osmond, Frémond and Cartaud, loc. cit.
H. M. Howe, loc. cit.
Hugh O'Neill, *Journal*, Iron and Steel Institute, Vol. 113, 1926, p. 417.

produced by a strain-anneal method. The details of the method and some peculiar results obtained with it will be reported in another paper. Some years of experience with the purification procedure has shown that the treatment used would reduce the carbon content to an amount that cannot be detected by the combustion method, the nitrogen and oxygen contents to below the limits of measurement by the vacuum fusion method, and also the sulphur to fairly low values. All purification treatments were carried out with elaborate precautions against contamination of the specimens from the surrounding refractories, using a furnace that will be described elsewhere. Any impurities remaining after this treatment could not be considered injurious for the present purposes.

The source and composition of the silicon alloys used are listed below.

Source	Analysis Silicon	Carbon	SiO_2
American Rolling Mill Company	1.13	0.014	0.054
	1.95	0.016	0.013
	3.42	0.012	0.009
	3.84	0.011	0.016
	4.17		
General Electric Company	3.3 ± 0.1		
	3.59		
Allegheny Steel Company	4.96	0.042	

The 1.13, 1.95, 3.42 and 3.84 per cent silicon alloys, in the form of bars from a ¼-inch thick plate, were purified in dry hydrogen for 10 days at 1340 degrees Cent. (2445 degrees Fahr.). During this time considerable grain growth took place, producing an average grain size of 5 millimeters. Purified bars of 1.95 and 3.84 per cent silicon were rolled from a thickness of ¼ inch down to 1/16 inch, and recrystallized to give grains from 5 to 15 millimeters in diameter.

The 4.17 per cent silicon sheet was a commercial product that had exhibited abnormal grain growth during the rolling and annealing process. Both purified and unpurified specimens of this material were used, but no difference was noted in their behavior. The purification consisted of a treatment of 100 hours at 1100 degrees Cent. (2010 degrees Fahr.) in dry hydrogen. Due to the thinness of the 4.17 per cent sheet this treatment was equivalent to that given the others.

The 3.3 and 3.59 per cent silicon alloys were strips containing large crystals which had been grown by W. E. Ruder of the General

Electric Company using the strain anneal technique. The specimens were from commercial heats and were unpurified.

The 4.96 per cent silicon alloy was from a commercial heat. It had been cast and cooled slowly in an ingot about 12 x 12 x 36 inches that had been covered with hot slag. When the ingot was fractured by impact, large crystals 15 millimeters in diameter were obtained; these were given no purification treatment.

A few of the iron and 4.96 per cent silicon crystals were found to contain occasional included crystallites. When it was necessary to use crystals with such inclusions, care was taken to confine observations to areas free from the crystallites. This precaution, together with a comparison of results from duplicate experiments on many different crystals, minimized the danger of spurious effects from this cause.

Experimental Procedure—Various methods of deformation were investigated in an attempt to evolve a method that would give reasonably straight slip lines. Cantilever bending and pressure figures made by pressure with a sharp point gave satisfactory results at times. Compression specimens deformed under loads slightly over the yield point were also used to some extent, but in view of the fact that much of the material on hand was in sheet form the compression method could not be used to advantage throughout the entire range of silicon content.

Torsion of thin rectangular single crystal specimens (one or more twists of a few degrees) gave by far the most satisfactory slip lines. Specimens of ½ inch to 1 inch long, ¼ inch wide, and about 1/32-1/16 inch thick were used in most of the torsion work. The deformation was small and relatively uniform, and produced slip on very few sets of planes, with remarkably straight slip lines in many instances. In certain crystals distorted by this method, only one system of parallel slip lines was found, a clear indication of stress homogeneity. The high temperature twisting was carried out in a furnace with a hydrogen atmosphere. The specimens were held in a jig and twisted from outside the furnace. Low temperature experiments were carried out in a Dewar flask equipped with a wooden specimen holder.

The angles made by slip, twin and cleavage traces on carefully polished specimens were measured with respect to reference lines. In the early part of the work the slip was photographed at a magnification of about 100 diameters with a cross hair in the field of view

parallel to the reference mark. Later the readings were made on a microscope using an ocular with cross hairs and a circular scale. A magnification of 70 was generally used, and angles were read to the nearest ½ degree.

The crystal orientations were obtained after the specimens had been deformed, using the back reflection Laue method and a 15-inch stereographic net.[26] In spite of the deformation, the X-ray films showed badly distorted spots, "asterism," only with warped specimens that had not been straightened after being twisted or bent. All of the crystals used were sufficiently perfect in their lattice structure to produce sharp diffraction spots in the undistorted state, though several strips of material on hand were discarded when they were found to be unsatisfactory in this respect.

After determining the orientation of a crystal and plotting the {1 1 0}, {1 1 2}, and {1 2 3} poles on the stereographic projection, taking the plane of the specimen as the plane of projection, the directions of traces observed on the specimen were plotted on the projection.*

This was done by drawing diameters perpendicular to the mean of the measured directions. Poles capable of having caused a given set of traces would then be found within a few degrees of the diameters so drawn, and such poles were entered in a table, together with the deviation between the predicted trace direction for each one and the mean direction that had been observed. When a plane predicted a set of traces more than 7 degrees away from the mean of the ob-

[26] A. B. Greninger, *Metals Technology*, American Institute of Mining and Metallurgical Engineers, Technical Publication 583, December 1934.

*The stereographic projection conveniently represents angle relations between crystal planes or their normals ("poles"). It may be described briefly thus: The crystal is assumed to lie at the center of a sphere, with the poles of its various planes intersecting the sphere as radii. The spherical surface with these intersection points is then projected, or mapped, on a plane (the "projection plane"). The method of projection is that which would occur if the sphere were transparent and a light located at the topmost point on its surface were to project markings on the surface of the sphere down onto a horizontal screen, the projection plane. Only the lower hemisphere need be projected. Angular relations may be measured by means of a stereographic net, which is the projection of the latitude and longitude circles of a sphere.

served traces, the plane was considered as definitely outside the limit of error of the method and was not considered as an operative slip plane in compiling the data. Similarly, the sets of measurements having a range of more than 10 degrees were thought to represent slip on two or more planes, or on a plane nearly parallel to the surface, and were not tabulated. Assigning definite limits of 7 and 10 degrees, respectively, to these tolerances was admittedly arbitrary, and a matter of personal opinion based on experience, but a considerable variation of these figures is possible without altering the conclusions. All sets of twin bands and cleavage cracks were measured and analyzed in the same way and served as a check on the accuracy of the orientation determination. The twin bands invariably showed less scatter in direction than the slip lines and always followed very closely the directions required of traces of the {1 1 2} planes. In fact, the average deviation of the predicted from the observed direction for 90 sets of twins on many different crystals was about 1 degree. It may be concluded, then, that the X-ray work and the stereographic manipulations were in error by about this amount, on the average, and that discrepancies much greater than this in the slip line analyses must be ascribed to the irregular nature of these lines rather than to experimental error.

Results—All of the sets of slip lines that could be measured with reasonable accuracy and whose measurements lay in a range of angles of less than about 10 degrees are entered in Tables I and II, along with the deformation conditions, the number of degrees range in the measurements of a single set of lines and the deviation of the mean of these measurements from the directions predicted for {1 1 0}, {1 1 2} and {1 2 3} planes—the three planes suspected of being slip planes in ferrite. When two or more of these planes could explain a given set of traces each was tabulated. Considering the large amount of previous work on this problem it seems entirely unnecessary to consider any other types of planes than {1 1 0}, {1 1 2} and {1 2 3}; confirming this is the fact that every trace tabulated—194 in all—could be explained by one or more planes of these three types.

In Figs. 1 to 5 are shown representative projections for some of the crystals included in the tables. These are drawn so as to show the range of angles for the slip lines of each set, the range appearing as an arc outside the graduated circle. The diameters in the projections are drawn to represent the predicted directions of the traces (actually both the arcs and the diameters are plotted perpendicular

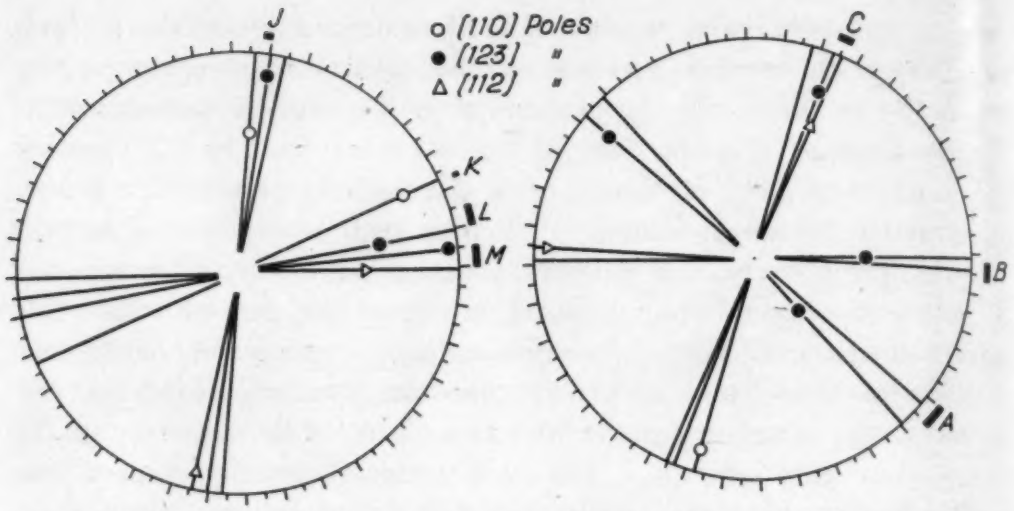

Fig. 1—Stereographic Analysis of Slip in Purified Iron at —195 Degrees Cent. Specimen 20; Photomicrograph Shown in Fig. 10. Diameters are Normal to Predicted Traces of Indicated Planes. Arcs Outside the Graduated Circle are Normals to the Observed Directions of Traces.

Fig. 2—Stereographic Analysis of Slip in 3.59 Per Cent Silicon Alloy at 20 Degrees Cent. Specimen 49. Diameters are Normal to Predicted Traces of Indicated Planes. Arcs Outside the Graduated Circle are Normals to the Observed Directions of Traces.

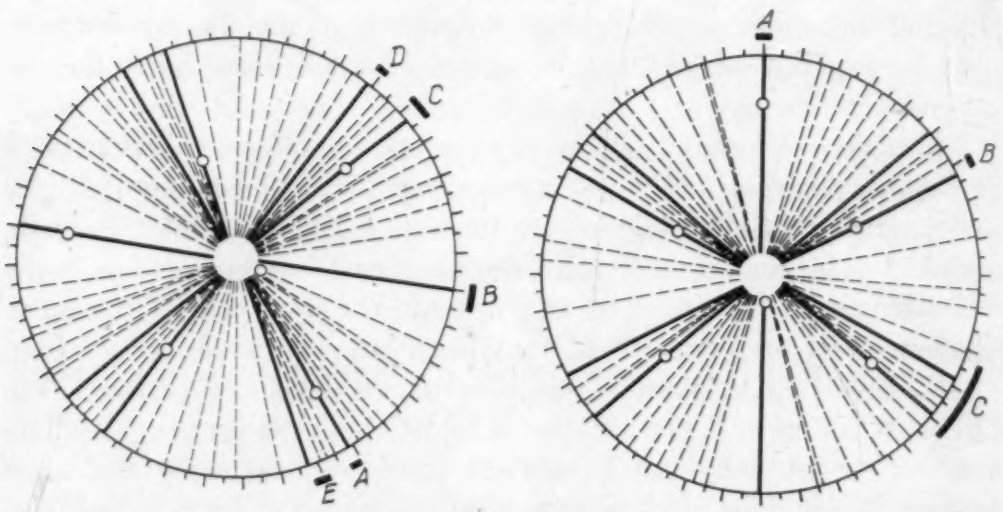

Fig. 3—Stereographic Analysis of Slip in 4.17 Per Cent Silicon Alloy at 20 Degrees Cent. Specimen 73. Arcs Outside the Graduated Circle Indicate Normals to the Observed Directions of Slip Lines; Small Open Circles Represent Poles of {1 1 0} Planes; Full Line Diameters are Normals to Predicted {1 1 0} Traces; Dashed Line Diameters are Normals to Predicted Traces of {1 1 2} and {1 2 3} Planes.

Fig. 4—Same as Fig. 3. Specimen 78.

to the traces on the specimens so that the diameters will pass through the poles of the corresponding planes). The designation of the traces (A, B, C, etc.) corresponds to the designation in Tables I and II. Missing letters in the alphabetical sequence merely indicate traces caused by twinning or cleavage—or in a few cases sets of slip lines

that were first measured but later found to be scattered over too large an angle to be of value in the analysis.

The conclusions to be drawn from Tables I and II are based on traces of two types; first, those traces for which a single plane of the suspected types is indicated, which instances are recorded in

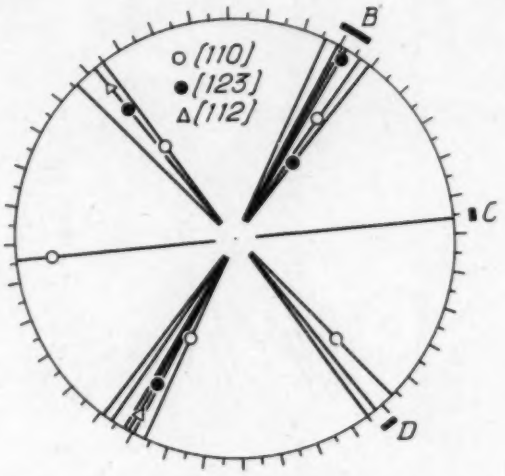

Fig. 5—Stereographic Analysis of Slip in 4.17 Per Cent Silicon Alloy at 535 Degrees Cent. Specimen 66. Photomicrograph Shown in Fig. 8. Diameters are Normal to Predicted Traces of Indicated Planes. Arcs Outside the Graduated Circle are Normals to the Observed Directions of Traces.

the final column of the tables, and second, those traces for which only two of the three planes are possible slip planes.

Table I shows that slip in iron is not on {1 1 0} planes alone, for 11 out of 45 sets of slip lines cannot be explained by {1 1 0} traces. Nor is it on {1 1 2} alone, for 24 out of the 45 cannot be correlated with {1 1 2} traces, nor on {1 2 3} alone since 14 sets are not accounted for in this way. It is necessary to postulate slip on {1 1 0} + {1 2 3} or on {1 1 0} + {1 1 2} + {1 2 3} to explain every trace. The slip mechanism is independent of temperature from 20 degrees to —195 degrees Cent.

Table II for various compositions of Fe-Si alloys shows similar results at certain compositions and temperatures. Referring to Fig. 6 where the results of Tables I and II are summarized, tests above and to the left of the curve require {1 1 0}, {1 1 2}, and {1 2 3} as planes of slip. There is no reason to believe there is any alteration of the slip mechanism throughout this entire area. Thus the mechanism {1 1 0} + {1 1 2} + {1 2 3} is indicated and this work con-

Table I
Slip Planes in Purified Iron

Specimen No.	Temperature of Deformation Deg. Cent.	Set of Slip Lines Marked	Scatter in Direction for Lines of Set	Deviation of Mean Direction from Direction Predicted by Plane of Form			Plane Uniquely Determined
				{1 1 0}	{1 1 2}	{1 2 3}	
1	20***	A	3°	0°	..	1°, 6°	
2	20***	A	4°	7°	2°	1°	
		B	2°	2°	5°	2°, 4°	
		C	3°	6°	1°	2°, 4°, 7°	
3	20*	A	3°	0°	..	4°	
		B	4°	..	3°	1°	
4	20*	A	5°	5°	..	..	(1 1 0)
5	20	B	3°	5°	4°	1°	
6	20	A	2°	6°	5°	4°	
		B	3°	1°	..	..	(1 1 0)
		C	0°	..	..	2°	(1 2 3)
7	20	A	2°	1°	..	4°	
		B	4°	..	0°	3°, 4°	
8	20	A	2°	2°	2°	0°	
		B	1°	..	1°	1°	
		C	4°	0°	..	5°	
		D	5°	4°	..	4°	
9	−70	A	1°	6°	..	0°, 2°	
10	−110	A	1°	1°	..	2°	
		B	1°	1°	..	..	(1 1 0)
11	−195**	K	8°	..	0°	4°	
12	−195**	J	10°	..	0°, 5°	3°	
		K	5°	..	5°	7°, 7°	
13	−195**	B	1°	1°	..	..	(1 1 0)
14	−195	F	0°	2°	3°	2°	
		G	7°	4°	..	..	(1 1 0)
		H	7°	7°	..	..	(1 1 0)
		I	2°	2°	..	..	(1 1 0)
15	−195	D	5°	2°, 7°	..	..	(1 1 0)
16	−195	G	4°	4°	2°	0°	
		H	0°	2½°	..	..	(1 1 0)
		I	0°	1°	3°	2°	
17	−195	E	4°	..	6°	1°, 1°	
		H	7°	..	5°	0°, 1°	
18	−195	F	2°	2°	..	2°	
		G	3°	3°	..	6°	
		H	3°	3°	..	..	(1 1 0)
19	−195	C	2°	4°	2°	..	
		D	8°	2°	..	..	(1 1 0)
20	−195	J	2°	3°	5°	1°	
		K	1°	1°	..	..	(1 1 0)
		L	5°	..	..	2½°	(1 2 3)
		M	5°	..	2½°	2½°	
21	−195	G	5°	4°	..	..	(1 1 0)
22	−195**	H	2°	..	..	2½°, 3°	(1 2 3)

*Deformed by bending in cantilever fashion.
**Deformed by pressing a sharp scribe into the surface.
***Deformed by compression.
(All other crystals deformed by torsion.)

firms Gough's conclusions as to the active slip planes in iron. Just why {1 1 2} traces occur *unequivocally* so rarely is uncertain—this is referred to in the discussion—but it should be noted that 36 out of 102 traces in this region *might* be {1 1 2} traces.

Below and to the right of the curve in Fig. 6 every trace is explicable as a {1 1 0} trace. A sufficient number were analyzed to make this conclusion amply justified (to make the probability of its being accidental very remote), but still another line of proof may be

Table II

Slip Planes in Silicon Ferrite

Specimen No.	Per Cent	Temperature of Deformation Deg. Cent.	Set of Slip Lines Marked	Scatter in Direction for Lines of Set	Deviation of Mean Direction from Direction Predicted by Plane of Form {1 1 0}	{1 1 2}	{1 2 3}	Plane Uniquely Determined
23	1.13	20***	A	5°	4°	4°	..	
24	1.13	20***	A	5°	1°	..	5°, 7°	
			B	3°	2°	..	2°, 5°	
25	1.13	20	A	0°	4°	..	1°	
			B	2°	1°	..	..	(1 1 0)
			C	2°	1°	..	0°, 2°	
26	1.13	−70	A	5°	2°	..	4°	
27	1.13	−110	A	2°	1°	..	4°	
			B	2°	1°	..	1°	
28	1.13	−110	A	2°	6°	..	3½°	
			B	3°	..	..	2°	(1 2 3)
			C	4°	3°	..	1°	
29	1.13	−195**	C	4°	0°	..	5°	
30	1.13	−195	F	2°	1°	..	2°	
			G	2°	1°	..	..	(1 1 0)
			H	2°	2°	..	0°	
31	1.13	−195**	D	3°	0°, 5°	5°	2°, 5°	
			F	4°	4°	..	..	(1 1 0)
			G	3°	2°	..	1°	
32	1.95	20	A	3°	..	1°	2°, 3°	
			B	0°	..	..	0°, 1°	(1 2 3)
33	1.95	−70	A	1°	5°	..	2°, 3°	
			B	1°	2°	..	2°	
34	1.95	−195	E	1°	1°	..	3°	
			F	1°	1°	..	..	(1 1 0)
35	1.95	−195**	G	1°	0°	..	..	(1 1 0)
			J	2°	2°	0°	..	
36	3.30	20	A	4°	..	..	1°	(1 2 3)
			B	4°	..	3°	2°	
37	3.30	20	A	2°	6°	..	..	(1 1 0)
			B	4°	2°	..	..	(1 1 0)
			C	0°	..	1°	3°	
38	3.30	0	A	5°	3°	..	..	(1 1 0)
			B	3°	..	1°	1°	
			C	1°	4°	..	1°	
39	3.30	−70	F	2°	3°	..	..	(1 1 0)
			G	4°	0°	..	1°	
			H	3°	0°	..	2°, 3°	
40	3.30	−70	F	3°	5°	3°	5°	
			G	4°	1°	..	..	(1 1 0)
41	3.30	−70	A	2°	1°	..	..	(1 1 0)
			B	2°	4°	..	3°, 3½°	
42	3.30	−110	C	2°	0°	..	..	(1 1 0)
43	3.30	−195**	F	1°	0°	4°	4°	
			D	3°	0°, 2°	3°	1°	
			F	3°	4°	..	1°, 4°	
44	3.30	−195**	J	3°	3½°, 4°	..	..	(1 1 0)
			K	2°	0°	..	2°, 3°	
45	3.30	−195	A	2°	1°	..	..	(1 1 0)
			B	1°	0°, 1°	2°	2°, 2°, 1°	
			C	1°	0°	..	4°	
46	3.30	−195	D	1°	2°	..	..	(1 1 0)
			E	4°	3°	..	4°	
47	3.59	500	A	1°	2°	..	..	(1 1 0)
			B	2°	4°	..	..	(1 1 0)
			C	1°	..	1½°, 1°	2°, 4°	
48	3.59	300	A	3°	6½°	..	2°	
			B	2°	1°	..	1°, 0°	

Table II (continued)

Specimen No.	Per Cent Si	Temperature of Deformation Deg. Cent.	Set of Slip Lines Marked	Scatter in Direction for Lines of Set	Deviation of Mean Direction from Direction Predicted by Plane of Form {1 1 0}	{1 1 2}	{1 2 3}	Plane Uniquely Determined
49	3.59	20	A	6°	..	..	½°	(1 2 3)
			B	4°	..	2°	3°	
			C	3°	7°	0°	2°	
50	3.59	−70	A	1°	3°	..	0°	
			B	1°	4°	..	2°	
			C	0°	1°	..	2°	
51	3.59	−70	A	2°	1°	..	1°, 2°	
			B	2°	..	..	4°	(1 2 3)
			C	4°	0°	2°	..	
52	3.59	−70	A	4°	3°	0°,0°	0°, 0°	
			B	1°	4°	..	..	(1 1 0)
53	3.59	−70	A	4°	1°	..	4°	
			B	3°	..	..	1°, 3°	(1 2 3)
			C	3°	1°	..	4°	
54	3.59	−110	C	2°	0°	..	..	(1 1 0)
55	3.59	−110	C	4°	1°	..	..	(1 1 0)
56	3.59	−195**	D	4°	0°	..	..	(1 1 0)
57	3.59	−195**	D	3°	0°	0°, 1°	..	
58	3.59	−195	F	3°	3°	..	..	(1 1 0)
59	3.84	20	A	3°	..	..	1°, 3°	(1 2 3)
			B	4°	..	1°	1°	
60	3.84	20*	A	2°	..	2°	..	(1 1 2)
61	3.84	0	A	2°	2°	..	..	(1 1 0)
			B	1°	..	2°, 3°	2°, 2°	
			C	2°	1°	..	..	(1 1 0)
62	3.84	−70	A	1°	7°	1°, 2°	..	
			B	1°	2°	..	1°, 3°	
			C	3°	..	6°	2°,2°	
63	3.84	−70	A	3°	1°	..	..	(1 1 0)
			B	1°	2°	..	..	(1 1 0)
			C	2°	1°	..	1°	
64	3.84	−195	F	2°	1°	..	..	(1 1 0)
			G	2°	1°	..	..	(1 1 0)
			H	2°	2°	..	4°	
65	4.17	700	A	4°	4°	..	..	(1 1 0)
66	4.17	535	B	8°	2°, 6°	1°	3°, 0°	
			C	3°	1°	..	..	(1 1 0)
			D	4°	3°, 6°	0°	0°	(1 1 0)
67	4.17	300	F	3°	1°	..	..	(1 1 0)
			H	7°	5°	3°	0°, 4°	
68	4.17	200	A	6°	1°	..	..	(1 1 0)
			B	3°	4°	..	1°	
			C	3°	1°	2°	1°	
			D	3°	0°	..	1°	
			E	4°	2°	..	..	(1 1 0)
69	4.17	20*	A	2°	2°	..	..	(1 1 0)
			B	4°	0°	..	..	(1 1 0)
			C	2°	1°	..	..	(1 1 0)
			D	5°	3°, 3°	2°	..	
70	4.17	20*	A	2°	0°, 3°	0°	..	
			B	3°	2°, 3°	3°	..	
			C	1°	2°	..	..	(1 1 0)
71	4.17	20*	B	2°	1°	..	..	(1 1 0)
			C	3°	2°	..	..	(1 1 0)
72	4.17	20*	A	3°	4°, 5°	4°	4°, 5°	
			B	6°	6°, 7°	2°	2°	

Table II (continued)

Specimen No.	Per Cent Si	Temperature of Deformation Deg. Cent.	Set of Slip Lines Marked	Scatter in Direction for Lines of Set	Deviation of Mean Direction from Direction Predicted by Plane of Form {1 1 0}	{1 1 2}	{1 2 3}	Plane Uniquely Determined
73	4.17	20*	A	3°	1°	..	3°, 5°	
			B	5°	2°	..	..	(1 1 0)
			C	6°	1°	..	4°	
			D	2°	1°	..	4°	
			E	4°	2°	..	3°	
74	4.17	20	A	2°	2°	..	..	(1 1 0)
			B	1°	4°	..	..	(1 1 0)
			C	4°	2°	..	1°	
75	4.17	20*	A	4°	0°	5°	2°, 3°	
			B	2°	2°	3°	1°	
76	4.17 (purified)	20*	A	1°	3°	..	..	(1 1 0)
			C	3°	2°	..	0°	
77	4.17	20	A	4°	0°	..	..	(1 1 0)
			B	3°	1°	..	4°	
78	4.17 (purified)	20*	A	3°	2°	3°	1°, 4°	
			B	3°	4°	..	2°	
			C	4°	5°	..	..	(1 1 0)
			D	2°	2°	..	..	(1 1 0)
79	4.17 (purified)	20*	A	1°	5°	2½°	0°, 2½°	
			B	1°	0°	4°	2°	
			C	1°	1°	..	..	(1 1 0)
80	4.17	−195	E	1°	2°	..	..	(1 1 0)
			F	3°	2°	..	3°	
81	4.17	−195**	F	little	3½°	..	1°	
			E	little	2°	3°	1°	
82	4.96	200**	A	2°	3°	4½°	..	
			B	3°	0°	..	..	(1 1 0)
			C	6°	1°	0°, 0°	..	
			D	7°	0°	..	..	(1 1 0)
			E	4°	1°	..	..	(1 1 0)
83	4.96	20**	A	7°	0°	..	..	(1 1 0)
			B	7°	0°	..	..	(1 1 0)
			C	7°	2°	1°	..	
84	4.96	20**	A	6°	0°	..	..	(1 1 0)
			B	4°	1°	..	..	(1 1 0)
			C	5°	0°	1°	..	

cited. The 4.17 per cent silicon crystals were bent about a number of different axes, in order to give many slip systems a chance to become operative. Yet even in these cases no planes other than {1 1 0} are required to explain the traces. This is well illustrated in the stereographic projections of Figs. 3 and 4, where slip lines would be expected lying parallel to nearly all the diameters shown, yet only the diameters drawn as full lines (predicted directions for {1 1 0} slip) are required by the actual measurements. Photomicrographs of this {1 1 0} slip are shown in Figs. 7 and 8.[27]

An interesting change in the microscopic appearance of the slip lines accompanies this change in mechanism. Ferrite deformed in the range of composition and temperature where all three slip planes

[27] A preliminary report of the work on this composition has been made. C. S. Barrett, *Physical Review*, Vol. 47, 1935, p. 809.

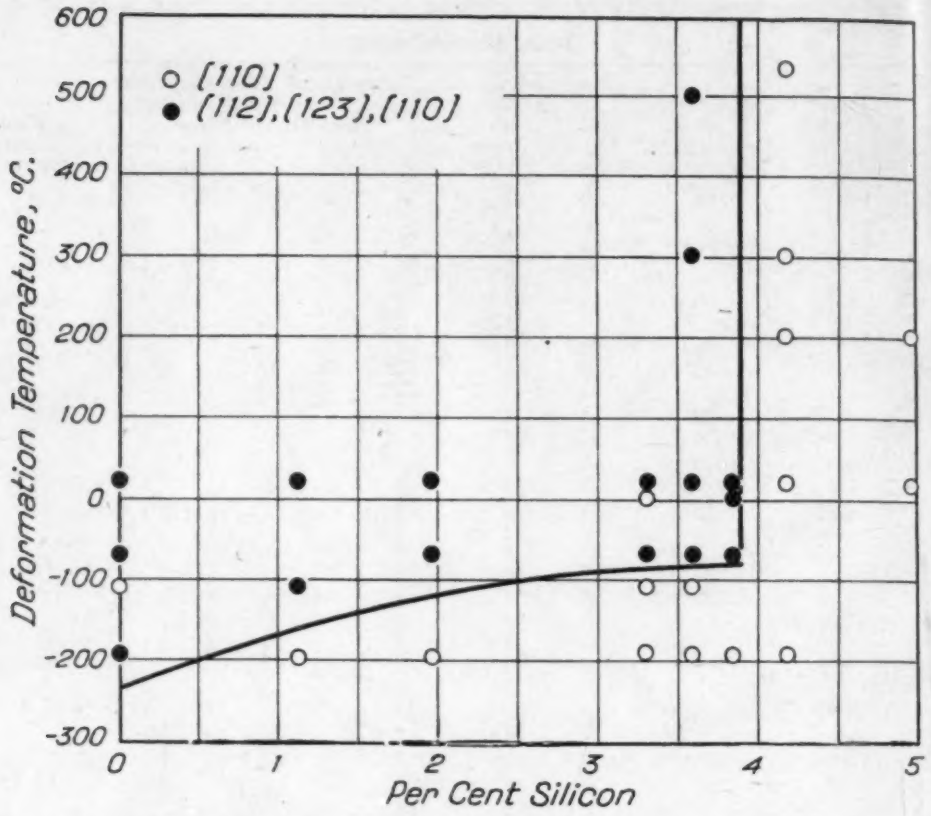

Fig. 6—Crystallographic Planes on Which Slip Occurs in Silicon Ferrite. Curve Indicates Change of Slip Mechanism.

operate exhibits lines that do not appear to be perfectly straight except in rare instances as, for example, at places where the homogeneity of stress is nearly perfect, or in cases where the slip direction lies parallel to the plane of polish (see Figs. 9 and 10). On the other hand, ferrite deformed in the range where only {1 1 0} slip occurs usually shows almost perfectly straight slip lines under all conditions of stress as, for example, around an indentation (see Fig. 11). The beautiful interlacing network characteristic of the thin torsion specimens in this range is almost unknown in the range of the normal ferrite mechanism (see Figs. 12 and 13). These characteristic differences may be accounted for on the basis of the multiplicity of the planes on which gliding takes place: when only the {1 1 0} planes are available for slip the change-over from one plane to another happens relatively infrequently and with a change of direction that is usually quite considerable in comparison with the normal mechanism.

Table III summarizes the data for the twinning plane in these

Table III
Twinning Planes in Iron and Silicon Ferrite

Composition	Temperature Degrees Cent.	Deformation	Number of Twin Directions	Average Deviation of Mean Direction from {1 1 2} Traces
Purified Iron	−195	Torsion	4	1°
Purified Iron	−195	Torsion	3	1°
Purified Iron	−195	Torsion	6	1°
Purified Iron	−195	Torsion	3	2°
Purified Iron	−195	Torsion	4	2°
Purified Iron	−195	Torsion	6	1°
Purified Iron	−195	Torsion	1	1°
Purified Iron	−195	Torsion	4	½°
Purified Iron	−195	Torsion	5	1°
1.13	−195	Torsion	5	1°
1.95	−195	Torsion	4	1°
3.30	−70	Torsion	1	2½°
3.30	−70	Torsion	5	1½°
3.30	−70	Torsion	5	1°
3.30	−110	Torsion	1	0°
3.30	−195	Torsion	3	1°
3.30	−195	Torsion	1	1°
3.59	−110	Torsion	2	½°
3.59	−110	Torsion	1	1°
3.59	−195	Torsion	6	1°
3.59	−195	Torsion	5	½°
3.84	−70	Torsion	(Directions not analyzed)	
3.84	−195	Torsion	5	2°
4.17	20	Impact	2	0°
4.17	20	Impact	1	1°
4.17	20	Tension	(Directions not analyzed)	
4.17	−70	Torsion	(Directions not analyzed)	
4.96	−70	Torsion	(Directions not analyzed)	
4.96	−70	Torsion	(Directions not analyzed)	
4.96	20	Impact	3	1°
4.96	20	Impact	4	2°

samples. In every instance the twins formed traces that agreed satisfactorily with the assumption of {1 1 2} as the twinning plane. As there has been no controversy on this point, and as the data were entirely consistent with previous determinations, no further discussion is required, but a sample projection is shown in Fig. 14 and photomicrographs in Fig. 15.

The plane of cleavage was determined in a similar way (see Figs. 16 and 17). Twenty-six cleavages of four different 4.17 per cent silicon crystals were analyzed, four cleavages in 4.96 per cent, three of 3.59 per cent, and three in purified iron. In every case the cleavage plane appeared to be {1 0 0} in agreement with previous determinations. (Low silicon alloys were cleaved at −195 degrees Cent., higher silicon alloys at −195 degrees Cent. and at +20 degrees Cent.)

B. On the Critical Stresses for Slip, Twinning, and Cleavage

Early experiments indicated that there is a threshold value of

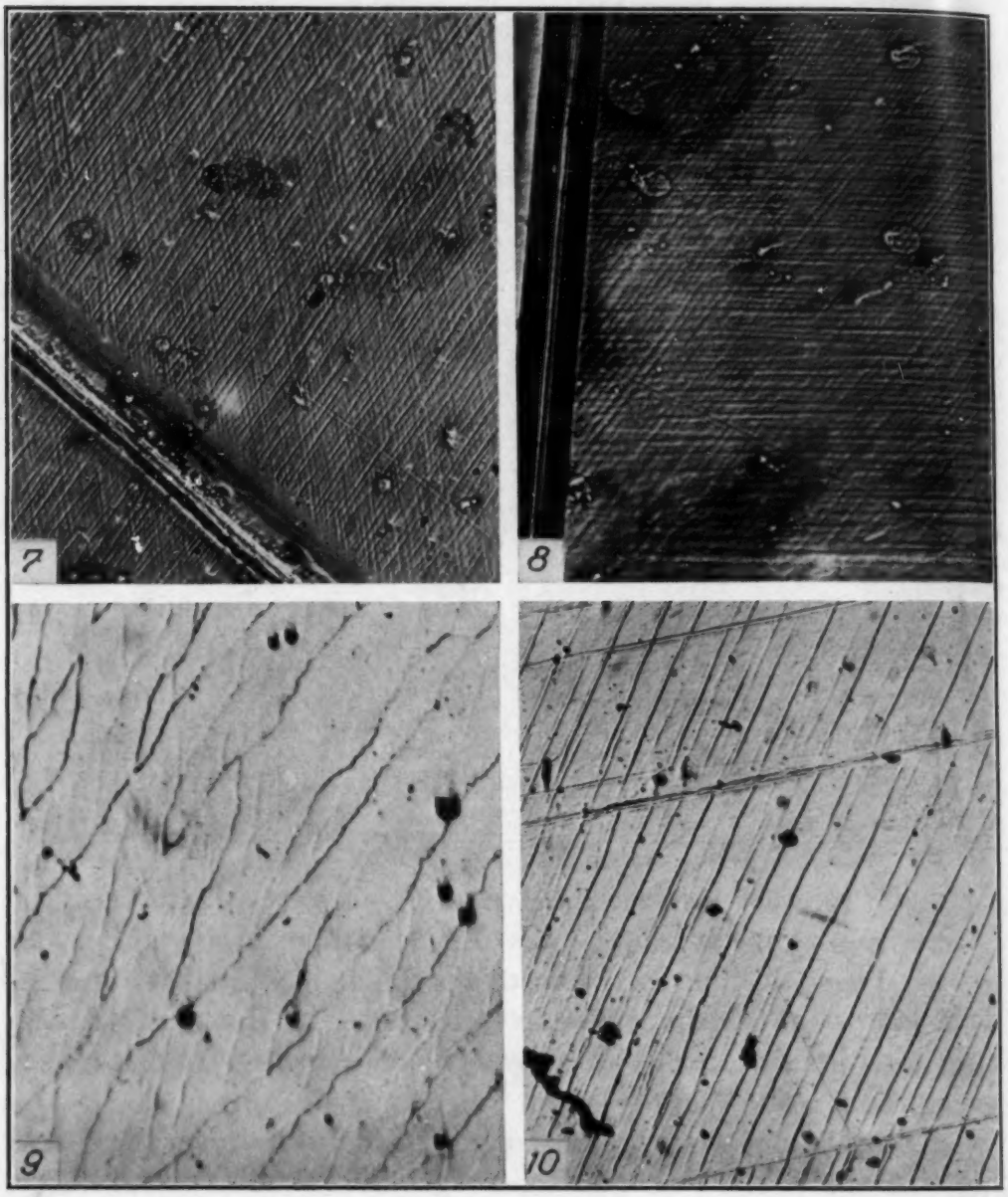

Fig. 7—Slip on {1 1 0} Planes in 4.17 Per Cent Silicon at 20 Degrees Cent. Portion of Reference Scratch Appears at Lower Left Corner.

Fig. 8—Slip on {1 1 0} Planes in 4.17 Per Cent Silicon at 535 Degrees Cent. × 100. Stereographic Analysis Shown in Fig. 5, Specimen 66. Portion of Reference Scratch Appears on Left. Slip Lines "B" and "C" Shown.

Fig. 9—Wavy Slip Lines in Purified Iron. Deformed by Compression at Room Temperature. × 200.

Fig. 10—Straight Slip Lines in Purified Iron. Deformed by Torsion at —195 Degrees Cent. × 100. Projection Corresponding to This is Fig. 1. Slip Lines in Field of View are {1 1 0} Traces (Set K, Specimen 20). Twins are Shown Running Horizontally.

shearing stress that must be exceeded before a crystal can deform by slip, a stress apparently independent of the normal stress acting

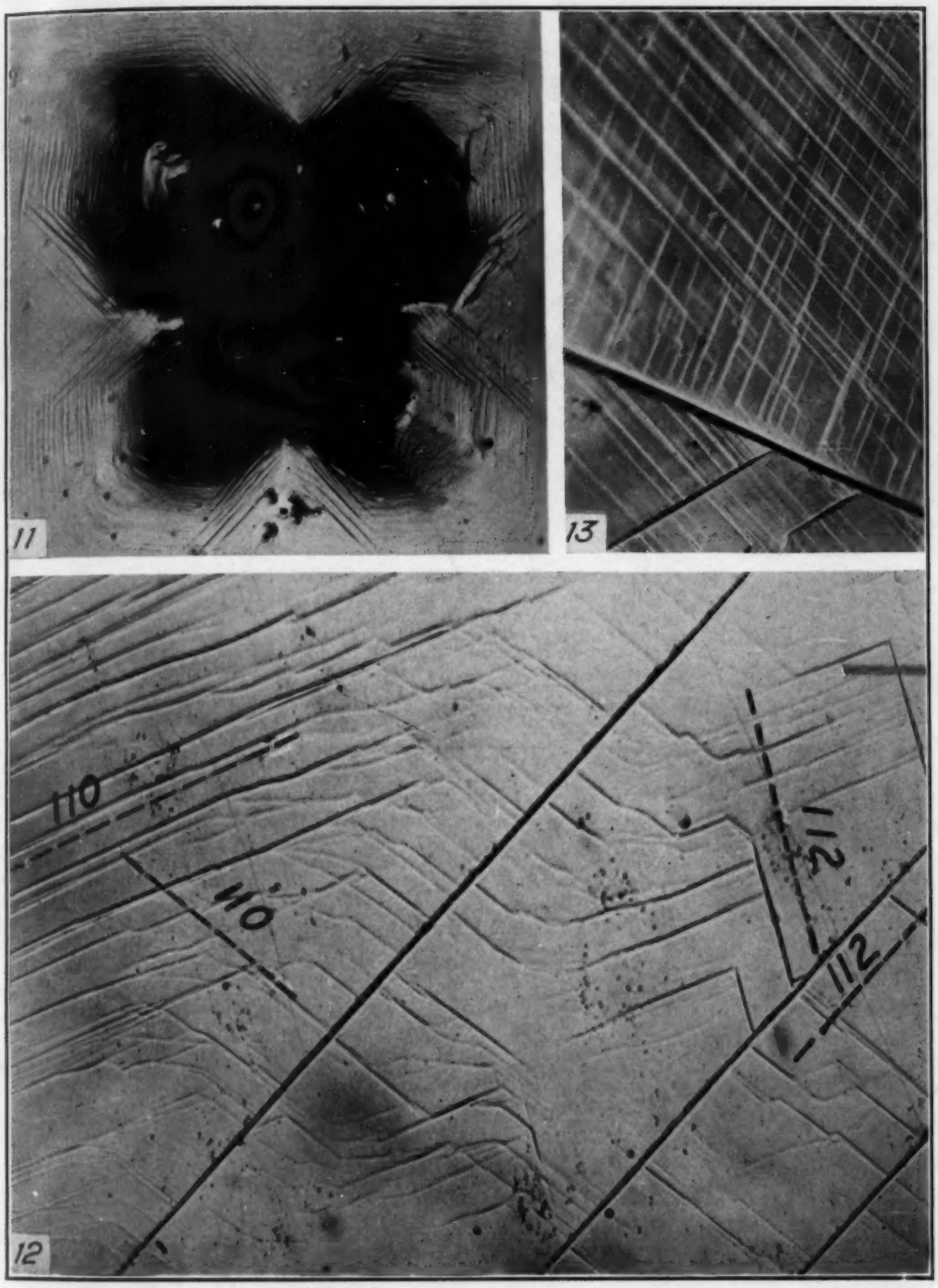

Fig. 11—Slip on {1 1 0} Planes Around a Pressure Figure in 4.96 Per Cent Silicon Alloy at 20 Degrees Cent. × 100. Polished Surface Parallel to Cube Plane.

Fig. 12—Twinning on {1 1 2} and Slip on {1 1 0} Planes in 3.84 Per Cent Silicon Alloy at —195 Degrees Cent. ×250.

Fig. 13—Same as Fig. 12, Near the Fracture. × 250. White Lines, {1 1 0} Slip; Black Lines {1 1 2} Twins.

across the slip plane.[28] The value of this shearing stress, resolved in the slip plane and in the slip direction, has been found to increase with decreasing temperature and it is also increased by solid solu-

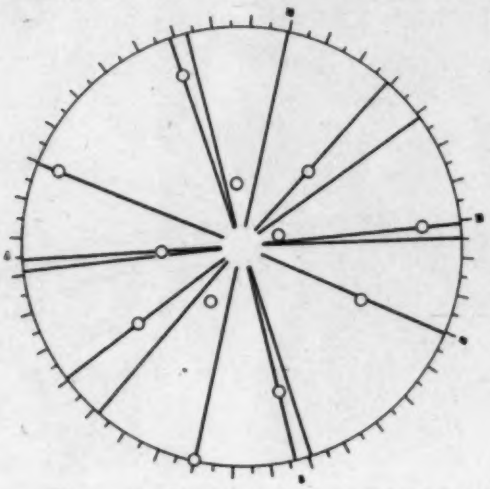

Fig. 14—Stereographic Analysis of Twinning in 4.17 Per Cent Silicon Alloy. Specimen Deformed by Impact at 20 Degrees Cent. Open Circles are Poles of {1 1 2} Planes; Diameters are Normals to Predicted Directions of Traces; Marks Outside the Graduated Circle Indicate Normals to Observed Directions of Traces.

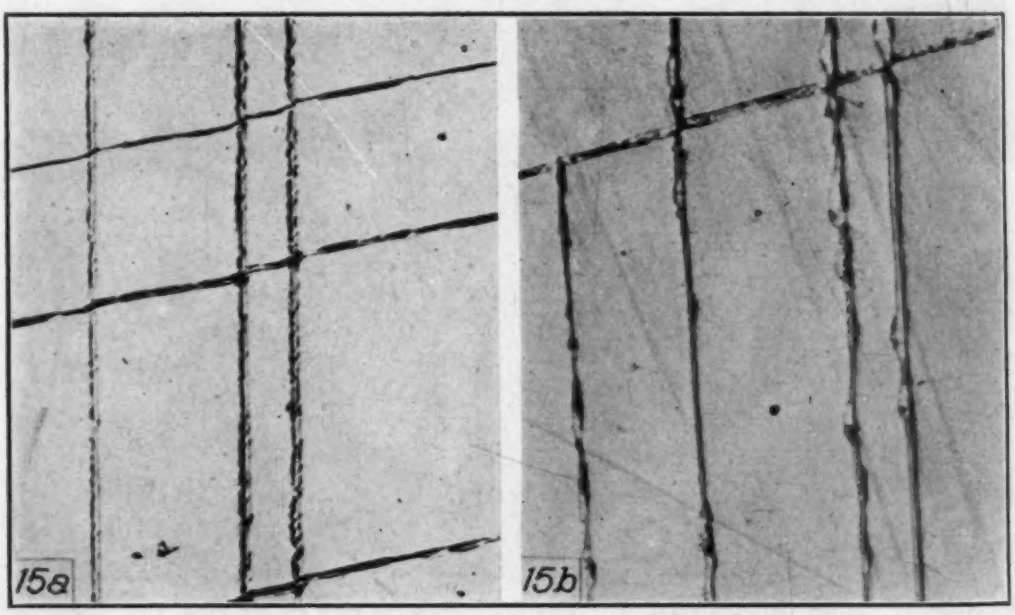

Fig. 15 a and b—Twins in 1.13 Per Cent Silicon Alloy, Produced by Torsion at —195 Degrees Cent. ×200.

Fig. 15a—Polished Before Twinning; Unetched.

Fig. 15b—Repolished Until Twins Disappeared; Then Etched in Nital. (Same crystal as 15a.)

[28]For a summary and references see E. Schmid and W. Boas, "Kristallplastizität," Springer, Berlin, 1934; International Conference on Physics, Vol. II, the Physical Society, London, 1935; C. F. Elam, "The Distortion of Metal Crystals," Oxford, 1935.

tion formation and by plastic deformation. One of the authors has emphasized the importance of creep tests in determining the existence of such a critical shearing stress and its magnitude,[29] and the general tendency of late is to regard such a critical stress as significant only as the stress at which the velocity of creep changes rapidly.[30] But

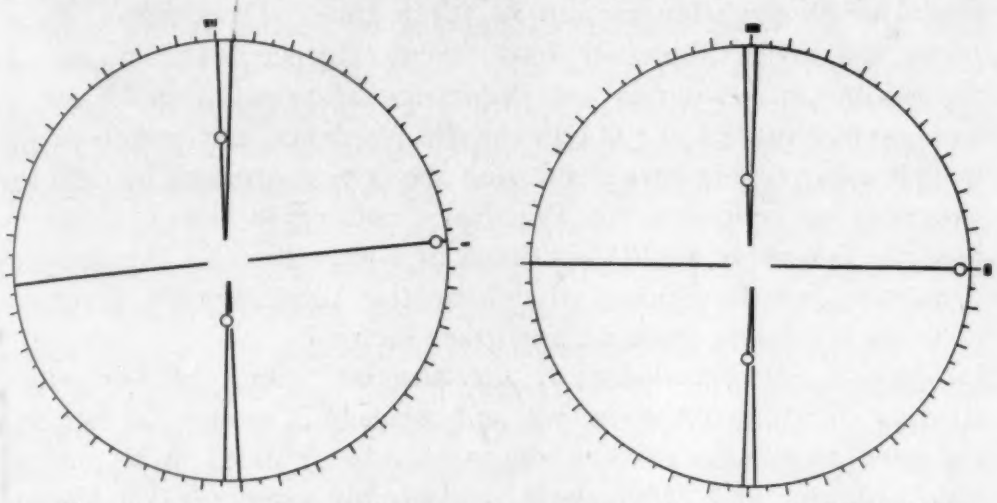

Fig. 16—Stereographic Analysis of Cleavage in 4.17 Per Cent Silicon Alloy. Specimen Cleaved at Room Temperature. Open Circles are Normals to {1 0 0} Planes; Diameters are Normal to Predicted Traces; Marks Outside the Graduated Circle Indicate Normals to Observed Traces of the Cleavage Faces.

Fig. 17—Similar to Fig. 16. 4.17 Per Cent Silicon Specimen Cleaved at 20 Degrees Cent.

regardless of these reservations as to the scientific significance of the critical shear stress for slip, it remains a concept of great value in understanding the ordinary mechanical behavior of stressed crystals.

The resolved normal stress required to cleave crystals is also a directly measurable quantity. In general, it is found to be but slightly influenced by temperature and by the amount of deformation that precedes cleavage, while the normal effect of elements added in solid solution is probably to increase the cohesion across cleavage planes, as cadmium does when dissolved in zinc.[31] In agreement with this is the observation that the internal pressure of a metal—a measure of its cohesion—is also slightly increased by solid solution formation.[32]

[29]R. F. Mehl, *Transactions*, American Institute of Mining and Metallurgical Engineers, Vol. 111, 1934, p. 144, in discussion to paper by Richard F. Miller.

[30]E. Orowan, *Zeitschrift für Physik*, Vol. 89, 1934, p. 605, 614, 634; Vol. 97, 1935, p. 573.
G. I. Taylor, *Proceedings*, Royal Society, London, Vol. A145, 1934, p. 362, 388, 405.
W. G. Burgers and J. M. Burgers, *Nature*, Vol. 135, 1935, p. 960; *Verhandelingen*, Royal Academy of Sciences at Amsterdam, Section I, Vol. 15, 1935, No. 3.

[31]E. Schmid, *Proceedings*, International Congress Applied Mechanics, Delft, 1924, 918.
E. Schmid, *Zeitschrift für Metallkunde*, Vol. 19, 154, 1925.
W. Fahrenhorst and E. Schmid, *Zeitschrift für Physik*, Vol. 64, 845, 1930.
F. Sauerwald and H. G. Sossinka, *Zeitschrift für Physik*, Vol. 82, 1933, p. 634.

[32]R. F. Mehl, Technical Publication 37, American Institute of Mining and Metallurgical Engineers, 1928.

Recent experiments[33] indicate that there is also a critical shearing stress associated with the twinning process. Whether this stress is the sole criterion for the beginning of twinning, or whether the normal stress across the twinning plane must also be considered, is still somewhat uncertain, and in view of the difficulties of measurement it will probably remain so for a time. Davidenkov, Kolesnikov and Fedrov, however, have found evidence for an increase in the resistance to twinning with decreasing temperature, in the case of zinc, up to a value 4 to 10 times the slip resistance, and have been able to plot approximate curves showing the critical stresses for slip and twinning vs. temperature. Practically nothing is known about the possible (probably slight) influence of composition of the twinning resistance, but the amount of deformation preceding the formation of twins is thought to be an important factor.

While our knowledge of the absolute values of the critical stresses initiating slip, twinning, and cleavage is meager, as has been indicated above, the *relative* values may be revealed in an indirect way. Maurer and Mailander[34] showed, for example, that the low temperature brittleness of iron is best explained by considering the ratio of the cohesion across cleavage planes to the slip resistance. This ratio decreases with decreasing temperature and eventually reaches a value that results in a brittle failure by cleavage in preference to a ductile failure by slip, the ductile failure having a fibrous or a silky appearance, the brittle fracture a granular appearance. In the vicinity of the critical temperature there is a transition region where the ductility is falling rapidly. The width of this transition region will depend on the angle at which the slip-resistance and cohesion curves approach each other; i.e., the rapidity with which the ratio of cohesion to slip resistance varies with temperature. Impact studies of alloy steels, for example, show a drop in impact that is gradual over a considerable temperature range and thus indicate a slow rate of change of the ratio of cohesion to slip resistance. Heindlhofer[35] used these concepts to correlate temperatures of cold

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[33]Davidenkov, Kolesnikov and Fedrov, *Journal*, Experimental and Theoretical Physics, (Russian), Vol. 3 (4), 350-360, 1933.

R. F. Miller, *Metals Technology*, Americal Institute of Mining and Metallurgical Engineers, Vol. 3, April 1936.

Bruce Chalmers, *Proceedings*, Physical Society, London, Vol. 47, 1935, p. 733.

H. J. Gough and H. L. Cox, *Proceedings*, Royal Society, London, Vol. A127, 1930, p. 431.

[34]E. Maurer and R. Mailander, *Stahl und Eisen*, Vol. 45, 1925, p. 409.

[35]K. Heindlhofer, *Metals Technology*, American Institute of Mining and Metallurgical Engineers, Vol. 1, Oct. 1934, (T. P. 581).

brittleness found in different types of test—notched bar impact, tensile, and torsion. The ratio of the greatest normal stress to the greatest shearing stress in a notched bar impact specimen is considerably greater than 2:1 (but is somewhat indefinite); in a tensile specimen it is 2:1, and in a torsion specimen it is 1:1. Therefore lowered plasticity occurs in the tensile test at a temperature where the ratio of cohesion to slip resistance is 2:1 (at about —155 degrees Cent. in iron) and where the ratio is 1:1 in the torsion test (below —185 degrees Cent. in iron). In the impact test the ratio exceeds 2:1 and induces brittleness at still higher temperatures (in the neighborhood of —20 degrees in iron). Since the cohesion of iron depends but little on the temperature, the resistance to flow must depend strongly on it.[36]

A similar argument may be used to explain the relative prominence of twinning and slip.[37] Twinning doubtless occurs only when the shearing stress in a suitable direction on a twinning plane exceeds the critical value, a circumstance that may be prevented by prior plastic yielding of the crystal on slip planes. The tendency of many metals, including iron, to twin more profusely as the temperature is lowered is thus an indication that slip resistance is increasing more rapidly than the critical stress for twinning.

The experiments reported below were designed to outline the area of the temperature-composition diagram in which silicon ferrite twins and cleaves during slow torsion. In the discussion following are presented schematic curves for these threshold stresses, which are consistent with the experiments reported, and which seem to integrate the data presented and to provide a clearer understanding of it.

Twinning in Single Crystals—Twinning was investigated in the same polished specimens that were used for slip determinations. The occurrence of twins was accompanied by audible clicking. Under the microscope the twins, as a rule, were quite easily distinguishable from slip lines for they had a definite width (or body) at high magnifications; this is shown, for example, in Fig. 15a; they are also visible after repolishing and etching, as in Fig. 15b. As previously men-

[36] F. Sauerwald, B. Schmidt and G. Kramer, *Zeitschrift für Physik*, Vol. 67, 1931, p. 179.

F. Sauerwald and H. G. Sossinka, *Zeitschrift für Physik*, Vol. 82, 1933, p. 634.

[37] C. H. Mathewson, *Proceedings*, American Institute of Mining and Metallurgical Engineers, Vol. 78, 1928, p. 7.

H. J. Gough and H. G. Cox, *Proceedings*, Royal Society, London, Vol. A127, 1930, p. 431. *Journal*, Institute of Metals, Vol. 48, 1932, p. 227.

E. Schmid and G. Wasserman, *Zeitschrift für Physik*, Vol. 48, 1928, p. 370.

International Conference on Physics, Vol. II, 1935, Paper by E. Schmid, and discussion of it by E. N. da C. Andrade.

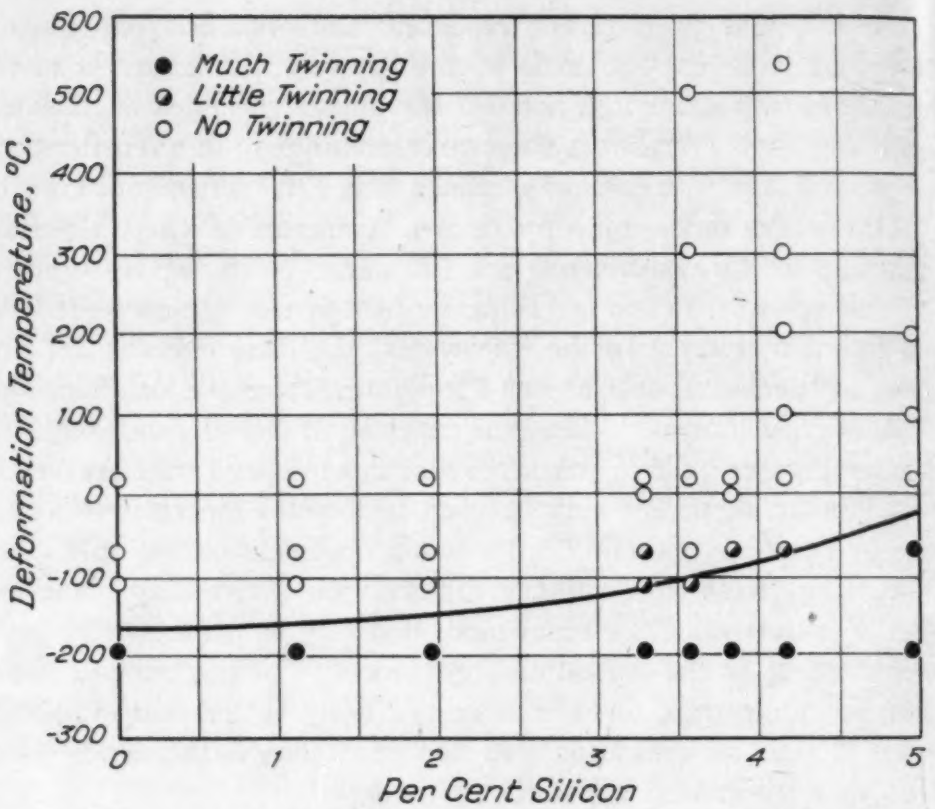

Fig. 18—Tendency for Twinning in Single Crystal Strips Under Slow Torsion.

tioned the predicted twin directions lay on the average within one degree of the observed twin directions and served as a check on the accuracy of the work.

Fig. 18 shows the temperature-composition region in which twinning is induced by static torsion in these single crystal strips. In constructing this curve only twins formed by slow torsion were considered, and twins obviously formed by the shock of cleavage during torsion were ignored, for in the latter case the stress conditions were not comparable to those that produced the principal slip lines.

Cleavage of Single Crystals—Cleavage of single crystals was studied as a function of temperature on specimens that previously had been analyzed for slip. The specimens were twisted through an angle of 45 degrees, and then the direction of twist was reversed to 45 degrees beyond the starting point (that is, the specimens underwent a 90-degree reversal). The number of reversals required to produce failure was counted. The data are presented in Fig. 19 in the following way. Specimens that withstood less than one cycle of

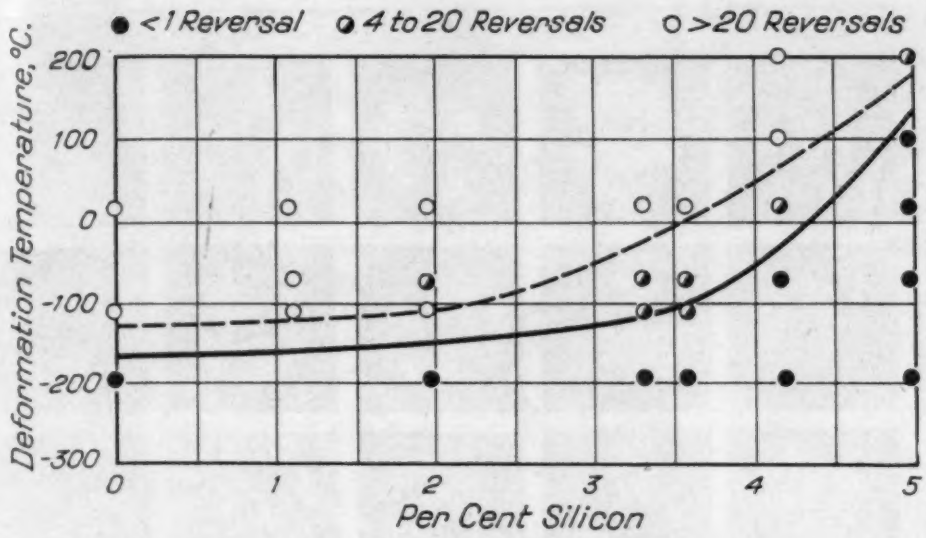

Fig. 19—Ductility of Single Crystal Strips in Slow Reversed Torsion. Full Line Represents Beginning of Brittleness; Dashed Line is Pilling's Curve of Brittleness for Rapid Bending of Sheets.

reversed torsion were plotted as brittle (filled circles); those withstanding 4 to 20 reversals were plotted as of intermediate ductility (half-filled circles); and those unbroken after 20 reversals were plotted as ductile (white circles). (Actually several crystals were studied under each set of conditions and the average behavior determined.)

Ductility of Polycrystalline Rods—Specimens for investigating the ductility of polycrystalline material as compared to single crystals were prepared from the purified 1.13, 1.95, 3.42, and 3.84 per cent silicon alloys. The specimens were swaged to a diameter of 0.132 inch and examined for internal faults, but none were found. There was also available some purified Armco iron, a special cut from the bottom of an ingot, that had received a lengthy purification treatment of 21 days above 1400 degrees Cent. (2550 degrees Fahr.) (See previous remarks on purity under "Materials and Treatment"). This also had been swaged to a diameter of 0.132 inch. Torsion specimens with a diameter of 0.075 inch over a gage length of ¾ inch and shoulders of 0.132-inch diameter were machined from the swaged rod. The ratio between specimen diameter and gage length diameter was chosen after experiments had shown that there was a likelihood that at low temperatures twisting would occur in the grips if the center portion were too large. After machining, the specimens were recrystallized at 700-800 degrees Cent. (1290-1470 degrees Fahr.).

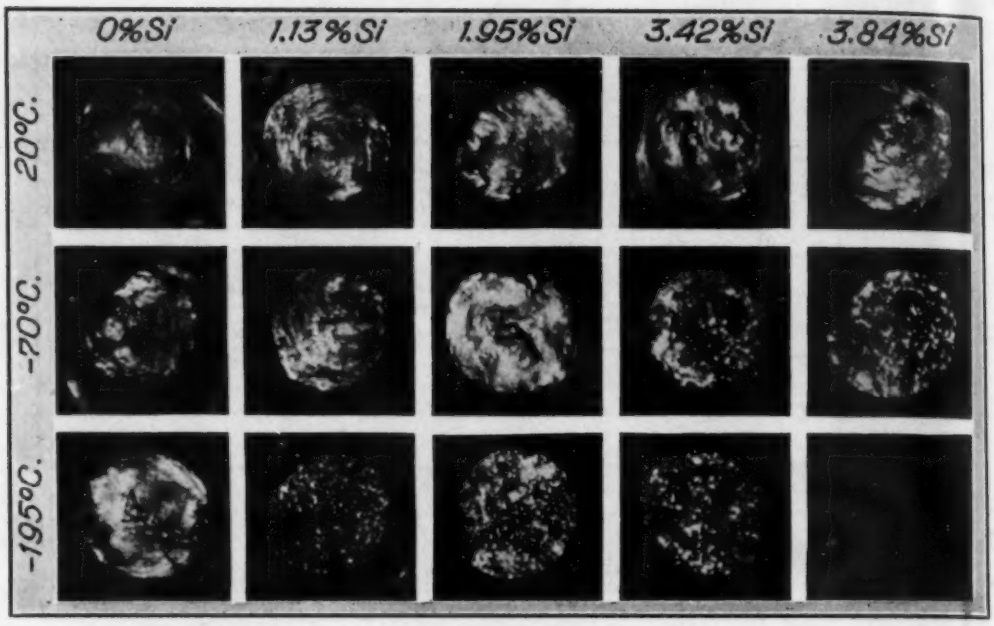

Fig. 20—Appearance of Fractures in Polycrystalline Torsion Specimens.

These torsion specimens when tested at low temperatures were immersed either in a mixture of solid CO_2 and acteone, or in liquid nitrogen in a metal container insulated with asbestos. Specimens were of sufficient length that the reduced section was at approximately uniform temperature. The rate of deformation was about two revolutions per minute.

A count was made of the number of revolutions required to produce failure, and the appearance of the fracture was noted. It will be seen from the photographs of the broken test pieces (Fig. 20) that the appearance of the fractured surface is capable of indicating clearly the course of the brittleness curve. The fractures were either completely granular, or completely smooth, fibrous and silky, or granular in one portion and silky in another. Reference to Fig. 21 will show that the brittleness curve plotted from the appearance of the break in this material parallels quite closely the curve for cleavage of single crystals despite the slightly different methods of stressing and criteria for plotting. Both curves for slow torsion resemble closely Pilling's ductility curve which was determined by bending strips in a vise.[38]

The number of turns before failure, however, was an unsatis-

[38]N. B. Pilling, *Transactions*, American Institute of Mining and Metallurgical Engineers, Vol. 69, 1923, p. 780-790.

factory indication of the course of the ductility curve: Completely granular fractures resulted from 3 complete revolutions at —195 degrees Cent. in alloys that gave smooth fractures at room temperature in 3 to 11 revolutions.

Discussion—With regard to the crystallographic mechanism of slip, the alloys studied in this series of experiments may be divided

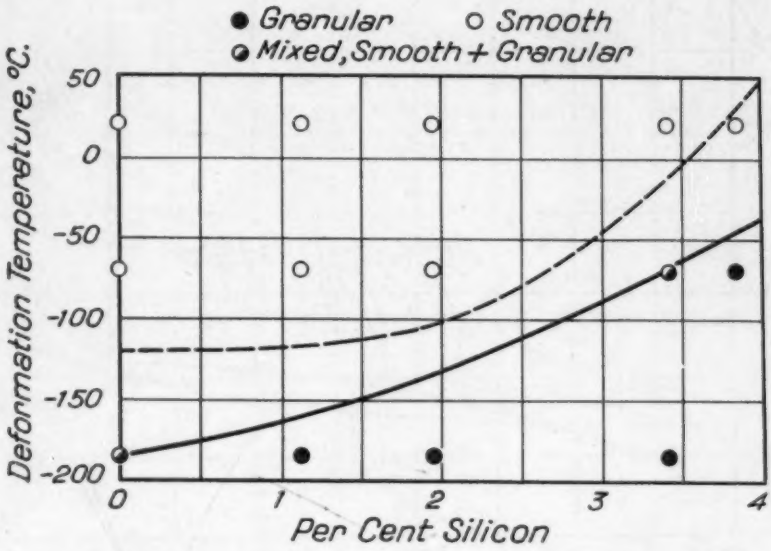

Fig. 21—Appearance of Fracture of Polycrystalline Torsion Specimens. Full Line Indicates Beginning of Brittle Type of Fracture; Dashed Line is Pilling's Curve of Brittleness for Rapid Bending of Sheets.

for convenience into three ranges with approximate limits of composition as follows: 0 to 1 per cent Si, with slip on {1 1 0}, {1 2 3} and {1 1 2} planes at all temperatures studied; 1 to 4 per cent Si, with slip at low temperatures on only {1 1 0}, and at higher temperatures on {1 1 2} and {1 2 3} also; 4 to 5 per cent silicon (or higher) with slip on {1 1 0} only at all temperatures investigated. The situation in these three ranges may be seen most readily by reference to Fig. 22 which summarizes the data of the preceding figures. It will be clear from the figure that the data are insufficient to decide whether or not iron changes its mechanism of slip at extremely low temperatures and should be classed with alloys of intermediate silicon content, but this question does not affect the following discussion.

If we now consider the curves of critical shear stress for slip on each of these planes as a function of temperature, we must conclude that in the low silicon range of composition the {1 1 0}, {1 2 3} and {1 1 2} curves are similar and nearly coincide at all temperatures

from +20 to —195 degrees Cent., for were they appreciably different, there would be slip on one plane to the exclusion of the others.* It should not be assumed, however, as seems to be the tendency of investigators in the field, that the slip resistance of these different planes is identical. In fact, the data in Tables I and II would suggest

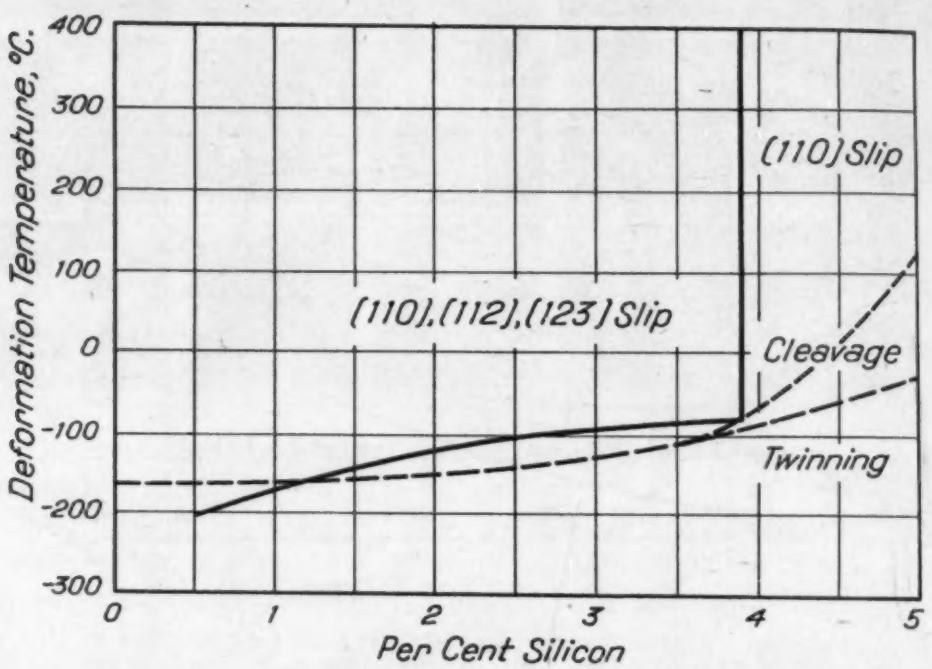

Fig. 22—Summary of Slip, Twinning, and Cleavage Data Presented in Preceding Figures. Full Line Separates Region of {1 1 0} + {1 1 2} + {1 2 3} Slip From Region of {1 1 0} Slip. Dashed Lines Give Upper Limits of Regions Where Cleavage and Twinning, Respectively, are Produced in Slow Deformation by Torsion.

that slip on {1 1 2} is somewhat more difficult than slip on {1 1 0} because the number of traces definitely caused by {1 1 0} greatly exceeds the number definitely caused by {1 1 2}, while with equal slip resistance, the {1 1 2} traces should number twice the {1 1 0} traces because the number of nonparallel sets of planes of the form {1 1 2} is twice the number of nonparallel sets of the form {1 1 0}. Additional data would be required, however, to prove this with any certainty, or to prove a similar relation between {1 2 3} and {1 1 0} slip.

In the intermediate range of composition it is necessary to postulate a separation of the {1 1 2} and {1 2 3} curves from the {1 1 0} curve at the lowest temperature where {1 1 2} and {1 2 3} slip occurs.

*There is an uncertainty in drawing these stress curves and in defining the limits of temperature and composition in which each crystallographic process operates, arising from the fact that observations were made on single crystals of differing orientation and therefore under differing resolutions of stress. Various considerations indicate that this orientation function is relatively unimportant in the present discussion.

This temperature varies from —150 to —90 degrees Cent. as the composition is increased from 1 to 3.84 per cent silicon. In the higher ranges of silicon content the {1 1 2} and {1 2 3} curves are separated at all temperatures from —195 to +535 degrees Cent. Fig. 23 has been drawn to correspond with these principles and with the general principle that slip resistance increases with decreasing temperature and with increasing alloy content in solid solution. The slip resistance curves are labeled S, the subscript giving the index of the slip plane.

At temperatures where the alloys are brittle, the normal stress across cleavage planes overcomes the cohesion of the lattice before the

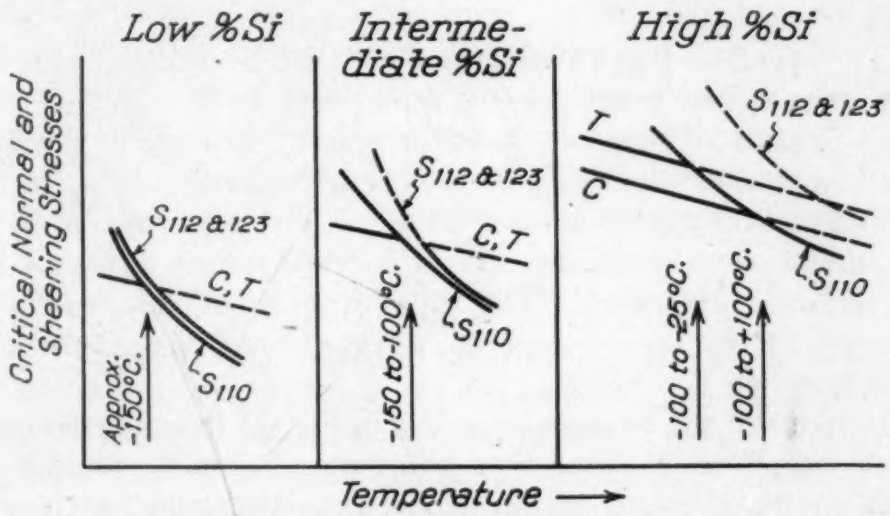

Fig. 23—Schematic Diagrams for the Variation of Critical Stresses for Cleavage (Curves C), Twinning (Curves T), and Slip (Curves S_{110}, S_{112} and S_{123}). Inhibited Processes are Indicated by Dashed Lines.

shearing stress overcomes slip resistance. At the upper limit of this temperature range of brittleness the slip resistance and cohesion curves must intersect. From the data presented in Fig. 22 it will be seen that the intersection must occur at about —150 degrees Cent. in low silicon alloys, slightly higher in alloys of intermediate composition, and from 0 to +100 degrees Cent. in the higher compositions of this study. The curves of normal stress for cleavage in Fig. 23 therefore have been drawn to intersect at approximately these temperatures the slip curve for the plane having minimum resistance to slip, for in the torsion test the maximum shear and normal stresses are equal. The curves of stress for cleavage, marked C in the figure, are drawn nearly horizontal, for the few available measurements on iron indicate

that the variation of this stress with temperature is only slightly, if at all, greater than the rather large experimental error.

That there is a critical stress for twinning and that it follows rather closely the curve for cleavage seems highly probable. In the lower ranges of composition these curves have been drawn coincident (there are no data available on the exact slope of the twinning curve in iron) in order to have them intersect the slip curve at the same point. As required by the data on twinning vs. composition, however, this concidence does not extend beyond about 3½ per cent silicon, and the curves of cleavage ("C") and twinning ("T") must cut the slip curve ("S_{110}") at different points, the twinning curve intersection being in the neighborhood of —50 degrees Cent. with 4 to 5 per cent silicon.

The curves in Fig. 23 are drawn with full lines to represent the active slip, twinning and cleavage processes in torsion and with dashed lines to represent inhibited processes. It must be admitted that the curves, as sketched, may have been elevated various amounts by strain hardening, and may not, therefore, represent the true conditions in undistorted materials. Yet it is felt that such errors are not very serious in this work. The effect of speed of deformation on the curves has not been studied, but further work is contemplated on this subject.

In terms of the concepts embodied in Fig. 23 then, the deformation characteristics of iron-silicon alloys may be readily understood. In this way the characteristic brittleness of high silicon alloys is to be regarded not as the result of an abnormal slip mechanism, in spite of the fact that room temperature brittleness occurs at roughly the same composition as does the abnormal slip mechanism, but as the result of the relative values of cohesion and slip resistance. The fact that silicon steels that are normally brittle can be rolled successfully at elevated temperatures is also consistent with this point of view, as the slip curve, S_{110}, always falls farther below the cleavage curve, C, as the temperature is increased. On the other hand, any precipitation from solid solution would increase the slip resistance—raise the S_{110} curve—and would thereby raise the point of intersection of the S_{110} and C curve (unless the C curve were raised as much as the S_{110} curve, a highly improbable circumstance).

Summary

Forty-five sets of slip lines in purified iron crystals have been

analyzed, using orientations determined by X-rays and stereographic projections. Slip in pure iron is on {1 1 0}, {1 1 2} and {1 2 3} planes and is unchanged from room temperature down to —195 degrees Cent. Slip lines in silicon ferrite (150 sets, in crystals up to 4.96 per cent silicon) show the same slip mechanism from +500 to —70 degrees Cent. and from 1 to 4 per cent silicon; but at higher percentages of silicon and at lower temperatures {1 1 2} and {1 2 3} slip is inhibited and only {1 1 0} slip remains. Mechanical twins ("Neumann bands") are formed on {1 1 2} planes under all conditions (there were 90 sets of these analyzed) and all cleavages (37 measurements) were on {1 0 0} planes. The change in slip mechanism does not coincide over quite the whole range of composition with the change in ductility and the beginning of twinning, both of which were measured as a function of temperature and composition. The relative stress requirements for slip, twinning and cleavage are plotted vs. temperature and composition in schematic diagrams based on these data, and are offered as a basis for understanding the variation in Fe-Si alloys of the brittleness and the tendency to twin.

Acknowledgments

The authors are indebted to Dr. Anson Hayes of the American Rolling Mill Company, W. E. Ruder of the General Electric Company, Dr. L. C. Hicks of the Allegheny Steel Company, and Dr. M. Gensamer of the Carnegie Institute of Technology, for their kindness in furnishing the material used in this investigation.

DISCUSSION

Written Discussion: By Alden B. Greninger, instructor in metallurgy, Harvard University.

The authors have indeed made a valuable contribution to our knowledge of the plastic deformation of alpha iron. It is believed, however, that an important characteristic of slip lines in alpha iron, i.e., their irregular nature, strikingly evident from the tabulated data, merits more attention than the authors have given it.

At first sight it would appear that little information could be gleaned from an attempt to match irregular traces with directions predicted ±7° by forty-two possible atomic planes. It must be conceded, however, that the authors have demonstrated a definite approximate relationship between slip lines and {1 1 0}, {1 1 2}, and {1 2 3} lattice planes. On the other hand, it must be admitted that these slip lines (seen at 70 magnification) show entirely too much irregularity—both in scatter and in deviation—for them to be regarded as true

traces of atomic planes. If we assume that slip movement is always parallel to an atomic plane, then we must conclude that most if not all the slip lines in ferrite, examined over distances of a hundred or more microns, must be a composite effect produced by minute displacements over alternate slip planes. Is it not possible that traces corresponding approximately with {1 1 2} or {1 2 3} are really composites of alternate slipping[1] on two or more {1 1 0} planes? All of these planes contain [1 1 1] directions, and each {1 1 2} plane bisects the angle between two {1 1 0} planes; likewise each {1 2 3} plane is located between a {1 1 0} and a {1 1 2} plane. Thus, if a slip were to proceed by approximately equal minute displacements along both $(1\,\bar{1}\,0)$ and $(0\,1\,\bar{1})$, the "slip line" might appear as an approximate $(1\,\bar{2}\,1)$ trace; if the relative displacements were 4:1 (instead of being equal) the "slip line" would be approximately $(2\,\bar{3}\,1)$. Variations in the relative amounts of slip along the different {1 1 0} planes could account for both the scatter and deviation found by the authors. In support of this hypothesis, we have the authors' statement that in the range where all three slip planes operate, straight slip lines are produced when (a) the homogeneity of stress is nearly perfect, and (b) the slip direction lies parallel to the plane of polish. Furthermore, the authors have found that, for high silicon contents, slip lines in ferrite are consistently rectilinear and all explicable (still, however, with both scatter and deviation) as {1 1 0} traces. If, again, we assume that slip in body-centered cubic metals is alternate {1 1 0} slip, then the behavior of high silicon ferrite roughly parallels the behavior of face-centered cubic alloys; it is well established that the major effect of the presence of alloying elements on the deformation of face-centered cubic crystals is the elimination of double slipping (the simultaneous slipping on two octahedral planes when the resolved shear stresses on these two planes are about equal).

It may be possible that very high magnification (U. V.) photomicrographs could establish whether the deformation of alpha iron proceeds in general by alternate {1 1 0} slip or whether the slip lines in this metal are really only approximately related to the lattice.

Authors' Reply

The suggestion of Dr. Greninger is identical with one recently proposed by Elam for slip in beta brass (which is also body-centered cubic); namely, that slip lines are formed by alternate slip on nonparallel {1 1 0} planes, and not by slip on any plane of higher index.[2] This alternate slip theory is contrary to a large amount of experimental data in the literature. Evidence in its support, both direct and indirect, is lacking, and furthermore it complicates rather than simplifies our conception of plastic flow because of the improbable assumptions it requires.

It should be noted that Dr. Greninger has not questioned that our conclusions are adequate to explain our data. As Tables I and II show, every set of traces that are straight enough to measure is in accord with our conclusions.

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[1]Various pictures of alternate slipping have been advanced previously by workers in this field, including Elam and Gough, in an attempt to explain the irregularity of slip lines in ferrite.

[2]C. F. Elam, "The Distortion of Metals," Clarendon Press, Oxford, 1935.

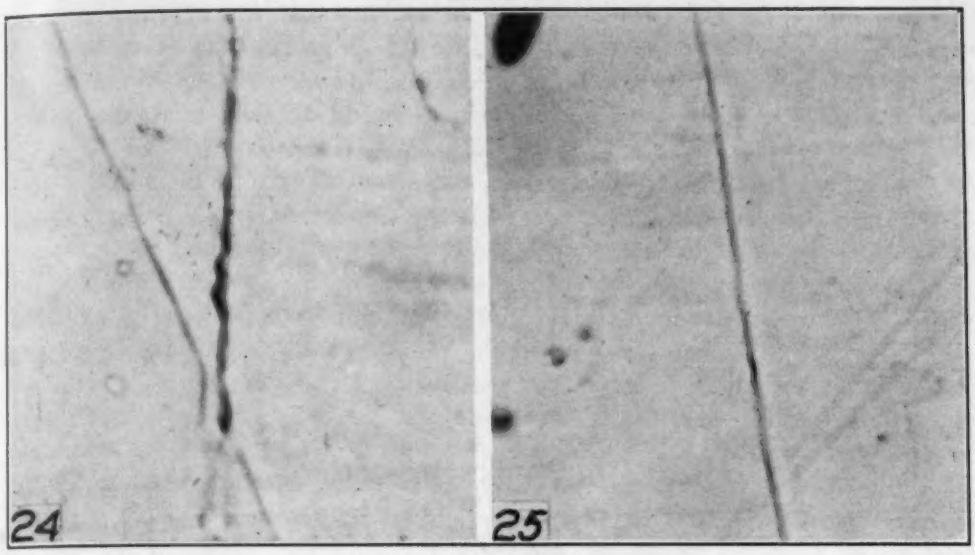


Fig. 24—Showing Pair of Slip Lines Produced in Iron at —195 Degrees Cent. × 2500.

Fig. 25—Slip in Same Specimen as Fig. 24 Lying Within 1 Degree of the Predicted Direction for a (1 2 3) Plane.

We have adequate reason to expect deviations from these directions because, as we frequently observe, slip does not take place on a single atomic plane but on different slightly displaced planes of a parallel set. We agree, of course, that the more irregular lines are the result of slip on nonparallel planes, but it is wholly unnecessary to limit these to planes of the {1 1 0} type as is assumed in the theory in question. Thus our point of view is adequate and Dr. Greninger merely suggests an alternate one because he thinks it simplifies matters. He states that it permits a correlation with certain double slip experiments on solid solutions, but, as to that, nothing we have found or stated is contrary to these same experiments.

The proposed theory would require duplex slip on planes along which the resolved stresses are greatly different. This is contrary to the vast amount of experimental data on single crystals, for the well established laws of slip in metal single crystals forbid duplex slip except when the resolved stresses are approximately equal. Gough's work has shown that even in cases of duplex slip it is possible to see the two components individually.

Another highly improbable assumption required by the theory is that the relative displacements on two planes must be in a definite ratio, such as 1:1 or 4:1, and must be the same ratio throughout the length of all the straight lines of a parallel set, even when the lines are found at widely separated points on the surface, as they frequently are.

Fig. 24 is a case that illustrates these objections to the theory. It shows a pair of slip lines produced in iron at —195 degrees Cent. with a magnification of 2500 diameters. At the left is a slip line caused by (1 1 0) slip; to the right of this and intersecting it is a slip line lying between calculated directions for two {1 1 2} planes, 2 degrees from one and 3 degrees from the other. On the theory we are discussing, the line on the left would be regarded as the trace of

a single atomic plane, and the one on the right would be regarded as having a saw-toothed structure throughout its length. In this particular instance, one edge of each saw tooth would be parallel to the line on the left and the other edge of each saw tooth would point in a direction 30 degrees to the right of the line that we see (since these are the nearest traces of {1 1 0} planes), and the zigzagging back and forth between these two directions would have to be uniform so that all along the line it would be made up of submicroscopic segments 62 per cent of one kind and 38 per cent of the other.

Fig. 25 is another slip line of the same specimen. It lies within 1 degree of the predicted direction for a (1 2 3) plane and so is adequately accounted for by our point of view. Yet, as in the previous figure, it would be regarded as having a saw-tooth structure on the theory Dr. Greninger mentions, with alternate saw-tooth edges lying at 56 degrees to each other and building up the observed line.

Thus while it is impossible to see what happens on a submicroscopic scale, we have shown at least that the theory has no microscopic support, that it is contrary to accepted principles, and that it complicates rather than simplifies our conception of the mechanism of slip.

EFFECT OF TITANIUM ON THE HARDNESS AND MICROSTRUCTURE OF HEAT TREATED 18 PER CENT CHROMIUM STEEL INGOTS

By R. E. Bannon

Abstract

This paper shows the effect of varying percentages of titanium on the size of grain and the amount of ingotism in cast 18 per cent chromium, 0.20-0.30 per cent carbon steel. In addition, the microstructure and hardness of a heat without titanium are compared with those of a heat containing 1.97 per cent titanium, after a series of heat treatments. The effect of titanium in eliminating air-hardening and hardenability is illustrated and evidence is shown of the solubility of the titanium constituent in the steel at 2500 degrees Fahr. Attempts were made to harden the steel by precipitating this constituent, but the results were negative.

Some changes of slope in the hardness curves are accounted for on the basis of expected changes in structure. Photomicrographs are included to illustrate some of the structures obtained by the various heat treatments.

THE work reported in this investigation was carried out using the bottom ends of ingots from four heats of high chromium steel. Three of the heats had been treated with varying amounts of titanium. The analyses of the four heats are given in Table I.

Table I
Analyses of Steels Used in this Investigation

	Per Cent				
	C	Cr	Mn	Si	Ti
Heat A	0.22	17.85	0.57	0.38	...
Heat B	0.30	17.75	0.70	0.91	0.25
Heat C	0.21	17.27	0.68	0.77	1.11
Heat D	0.22	17.65	0.69	0.77	1.97

Macrographs of these four ingots are shown in Figs. 1 to 4. Alloys C and D were etched with equal parts of aqua regia and glycerine, a reagent proposed by Vilella. Samples A and B, while attacked

A paper presented before the Eighteenth Annual Convention of the Society held in Cleveland, October 19 to 23, 1936. The author, R. E. Bannon, is research associate, The Titanium Alloy Manufacturing Co., Niagara Falls, N. Y. Manuscript received June 2, 1936.

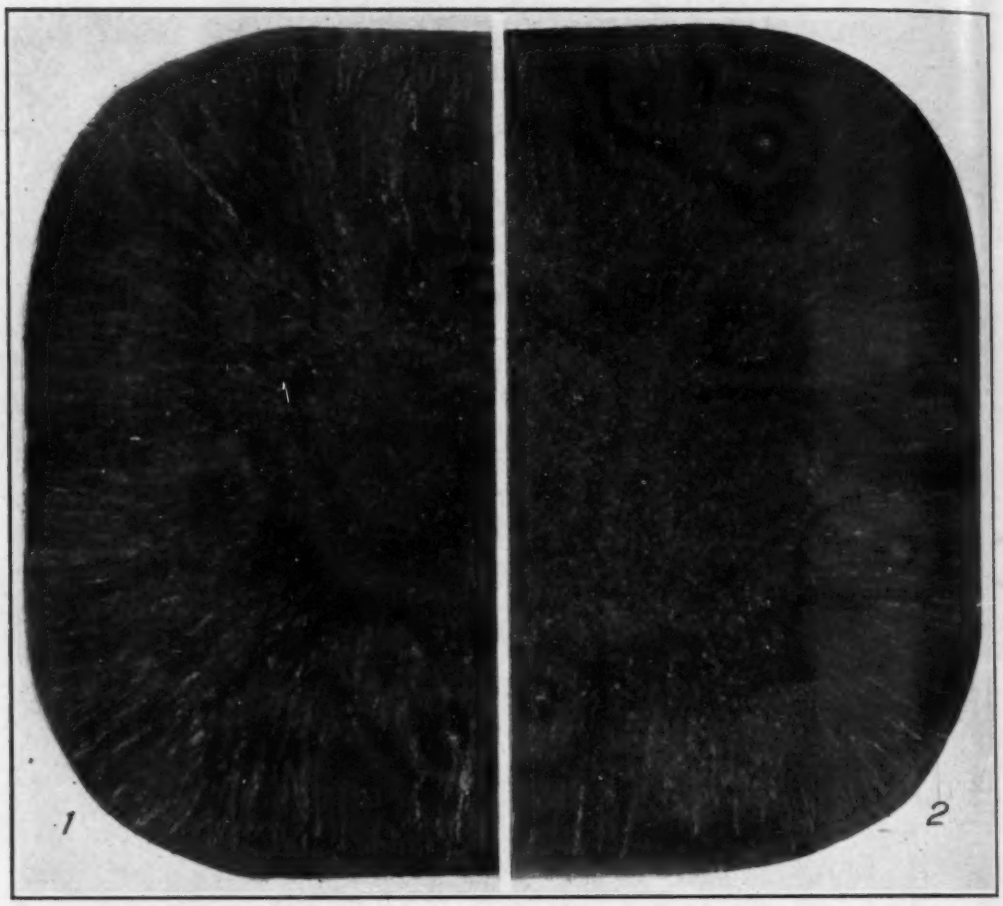


Fig. 1—Alloy A Ingot Section As-Cast, Showing Pronounced Ingotism. Etched with "Chrome Regia." × 1.7.

Fig. 2—Alloy B Ingot Section As-Cast, 0.25 Per Cent Titanium Showing Less Pronounced Ingotism. Etched with "Chrome Regia." × 1.7.

by this reagent, failed to show the structures clearly enough. The "chrome regia" proposed by Newell—5 parts of concentrated hydrochloric acid to 4 parts of 10 per cent chromic acid—was the best of a number tried, although the structures are not as clear as those shown in alloys C and D.

These macrographs are of particular interest in that they show a very marked refinement of grain size with increase of titanium content. Furthermore, with sufficiently high titanium there is an almost complete elimination of columnar grains except at the very edge. This is illustrated strikingly in Figs. 3 and 4. In those applications where working of the steel is not practical this elimination of ingotism may be of considerable importance.

In order to study the effect of titanium on the microstructure and hardness of this type of steel, the following heat treatment pro-

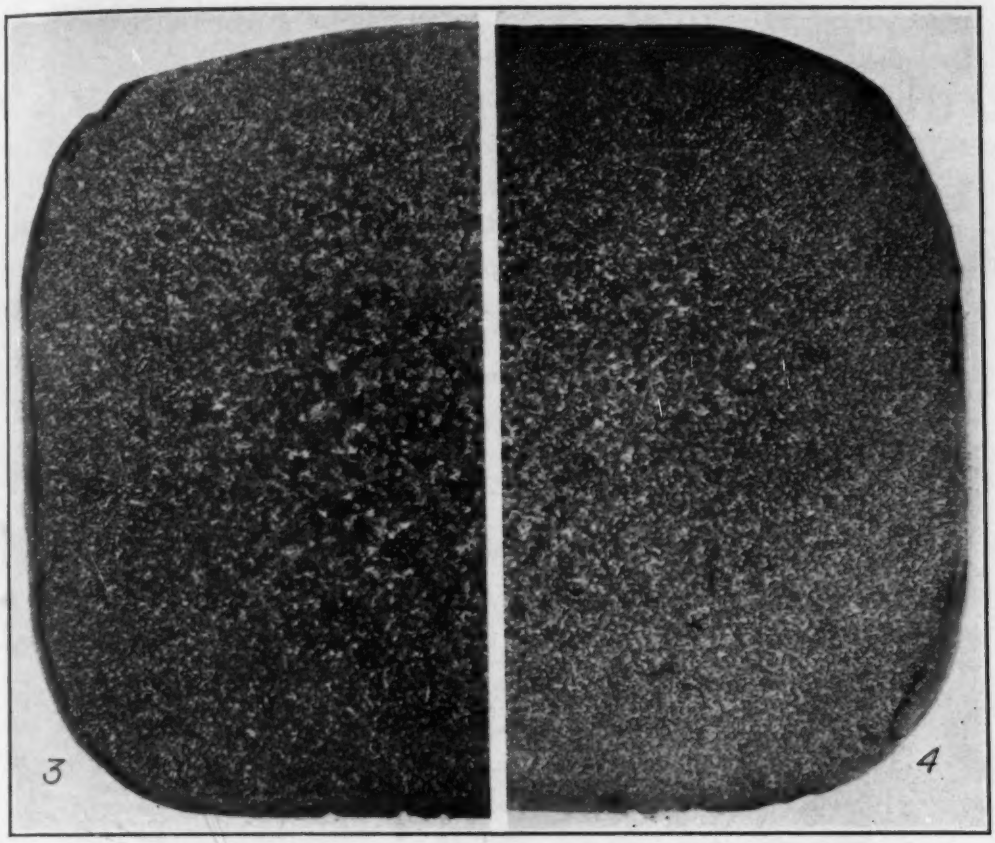


Fig. 3—Alloy C Ingot Section As-Cast, 1.11 Per Cent Titanium Showing Fine-Grained Structure with Ingotism Practically Eliminated. Etched with Aqua Regia in Glycerine. × 1.6.

Fig. 4—Alloy D Ingot Section As-Cast, 1.97 Per Cent Titanium Showing Finer Grained Structure with Ingotism Practically Eliminated. Etched with Aqua Regia in Glycerine. × 1.6.

gram was carried out on steels A and D, the latter being selected as having the highest titanium content of the series:

Heat Treatment Schedule

Water-quench from 2100 degrees Fahr.
Water-quench from 2100 degrees Fahr. and draw at 1650 degrees Fahr. for 5 hours.
Water-quench from 2250 degrees Fahr.
Water-quench from 2250 degrees Fahr. and draw at 1650 degrees Fahr. for 5 hours.
Water-quench from 2400 degrees Fahr.
Water-quench from 2400 degrees Fahr. and draw at 1650 degrees Fahr. for 5 hours.
Water-quench from 2500 degrees Fahr.
Water-quench from 2500 degrees Fahr. and draw at 1650 degrees Fahr. for 5 hours.

All samples drawn were furnace-cooled from the drawing temperature.

The heating for quenching was done in a gas-fired furnace. The specimens were protected from the flame inside a graphite crucible, so that a reducing atmosphere was maintained. The temperature inside this crucible was first brought up to that desired, the specimens were placed in it and held for 1 hour after they had reached the

proper temperature. The drawing was done in an automatically-controlled resistance furnace.

Ingot A was annealed in the same furnace for 3 hours at 1650 degrees Fahr. (900 degrees Cent.) and furnace-cooled in order to facilitate cutting up to obtain specimens for the above treatments.

Rockwell B hardness determinations were made after the treatments described and also on the ingots as cast. A section of ingot D was annealed for 3 hours at 1650 degrees Fahr. (900 degrees

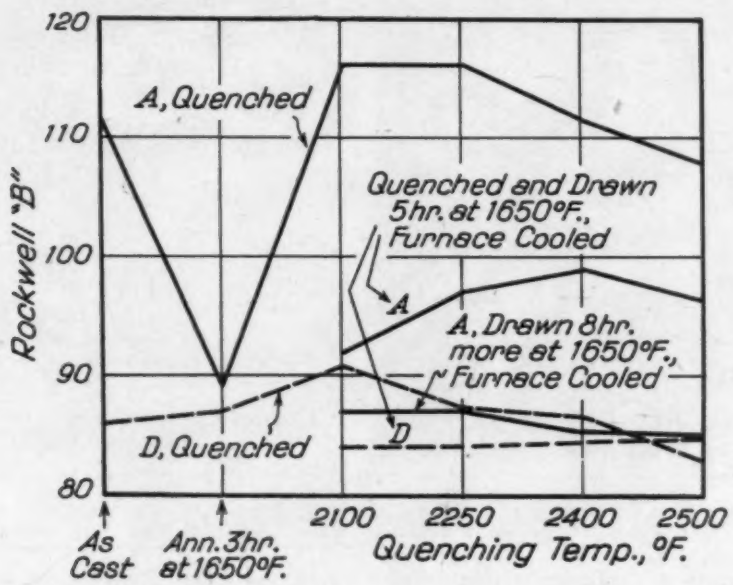


Fig. 5—Results of Rockwell B Hardness Tests Comparing Alloys A and D in the Heat Treated Condition.

Cent.) and furnace-cooled. The hardness of this was taken for comparison with the annealed sample from ingot A. The results of these hardness tests are shown in the diagram in Fig. 5.

Sections were taken for microscopic examination from all samples from ingot D — as cast, quenched and those subsequently drawn. Sections from ingot A examined included the annealed sample and those that had been drawn after quenching.

Figs. 7 to 10 illustrate the appearance of the microsections of alloy A after etching. Before proceeding with a discussion of these, reference should be made to Fig. 6, a reproduction from Krivobok's[1] cross section diagram for iron-carbon alloys with 18 per cent chromium. Line A-A represents an alloy of the same carbon content as

[1]V. N. Krivobok, "The Book of Stainless Steels"—published by the American Society for Metals, 1935, p. 34.

ingot A of this series. At temperatures of 2100 and 2250 degrees Fahr. (1150-1230 degrees Cent.) the alloy was just about at the boundary of the all-gamma and the gamma plus delta fields. The structure shown in Fig. 8 is representative both of the sample quenched from 2100 degrees Fahr. (1150 degrees Cent.) and also of that quenched from 2250 degrees Fahr. (1230 degrees Cent.). Apparently, judging from this photomicrograph, the samples were in

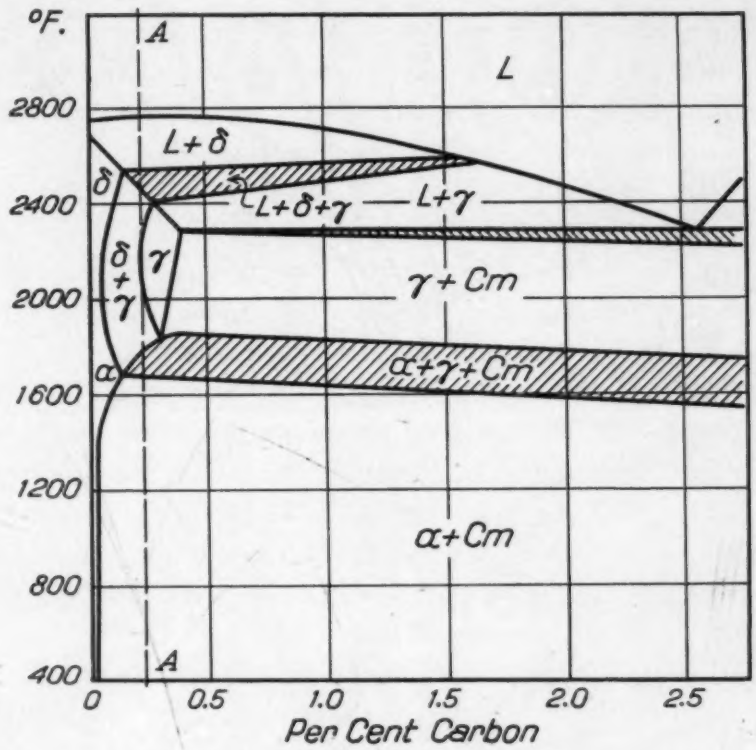


Fig. 6—Cross Section Diagram of Iron-Carbon Alloys Containing 18 Per Cent Chromium.

the gamma plus delta field at 2100 and 2250 degrees Fahr. (1150-1230 degrees Cent). At 2400 degrees Fahr. (1315 degrees Cent.) the alloy is also in the gamma plus delta field, with appreciably more delta. The subsequent quenching resulted in a lower hardness value and, as seen in Fig. 9, a larger proportion of ferrite — delta at the higher temperature.

Since a lowering of the chromium content causes a raising of the upper limit of the gamma plus delta field (Fig. 6), it is difficult to show exactly the state of alloy A at 2500 degrees Fahr. (1370 degrees Cent.). No evidence was noted that partial fusion had taken place and it seems logical to assume that at this temperature the al-

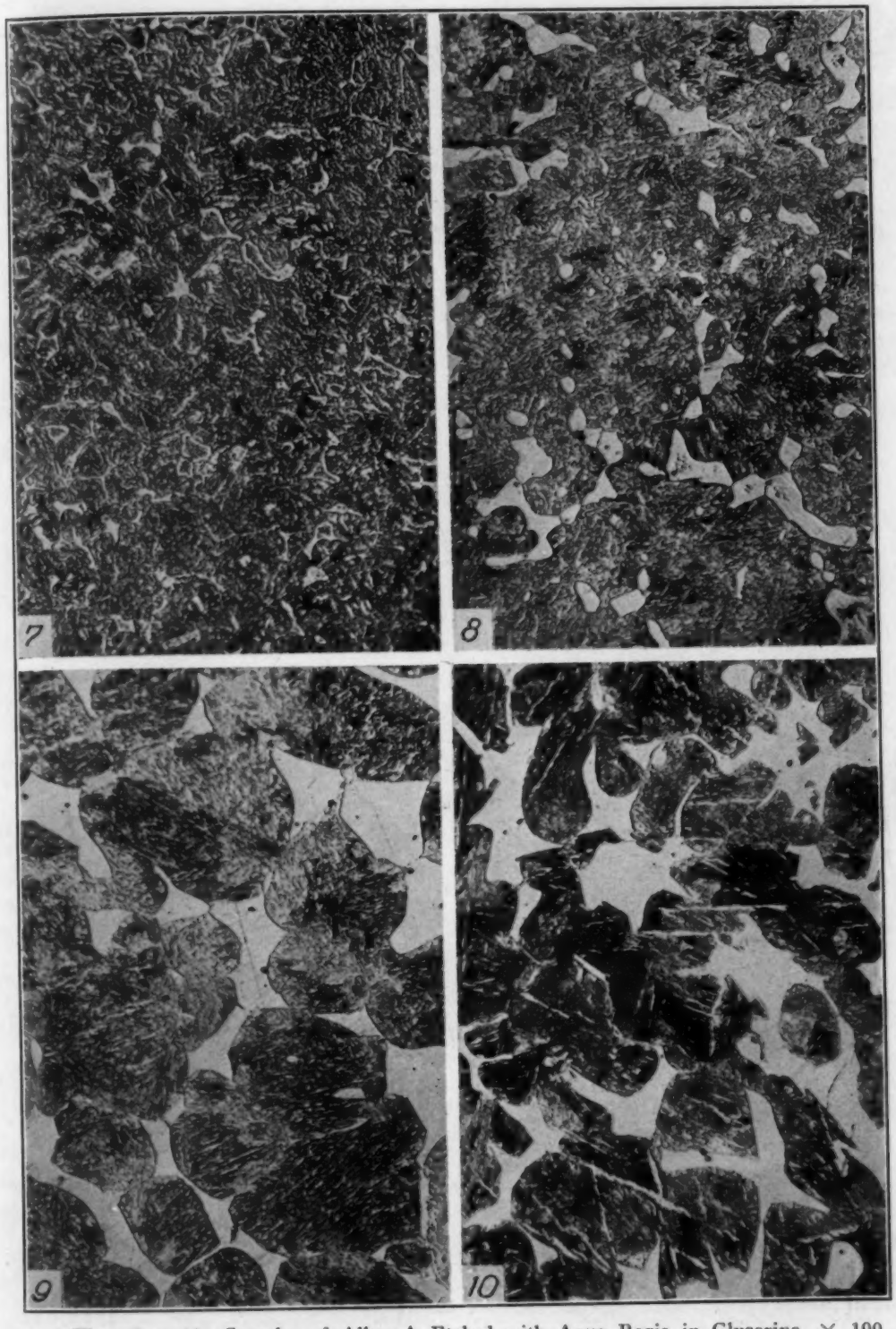
loy consisted also of gamma plus delta, with a greater percentage of delta than was present at 2400 degrees Fahr. (1315 degrees Cent.). This would result in a still lower hardness value on quenching, a fact borne out in Fig. 5.

The shape of the hardness curve for the samples from this alloy which had been drawn for 5 hours made it doubtful that a sufficient length of time had been used. The greater hardness of the samples quenched from 2250 and 2400 degrees Fahr. (1230-1315 degrees Cent.) and a lowering of hardness for the one quenched from 2500 degrees Fahr. (1370 degrees Cent.) (while still harder than the 2100 degrees Fahr. sample) may be accounted for by the following explanation. The samples quenched from 2250, 2400 and 2500 degrees Fahr. (1230, 1315, 1370 degrees Cent.) probably had successively more austenite and in the decomposition of this and the martensite also present, during the 5 hours drawing, less complete transformations probably took place in the samples quenched from the higher temperatures. The samples quenched from 2100 and 2250 degrees Fahr. (1150-1230 degrees Cent.) had the same hardness value (Fig. 5). The composite hardness of the mixtures of austenite and martensite present were the same, but after drawing for 5 hours at 1650 degrees Fahr. (900 degrees Cent.), followed by furnace-cooling, the greater amount of transformation that took place in the sample treated at the higher temperature resulted in a harder matrix. The larger proportion of ferrite in the 2400 and 2500 degrees Fahr. (1315-1370 degrees Cent.) samples could off-set, to some extent, the greater hardness of the matrix in these samples.

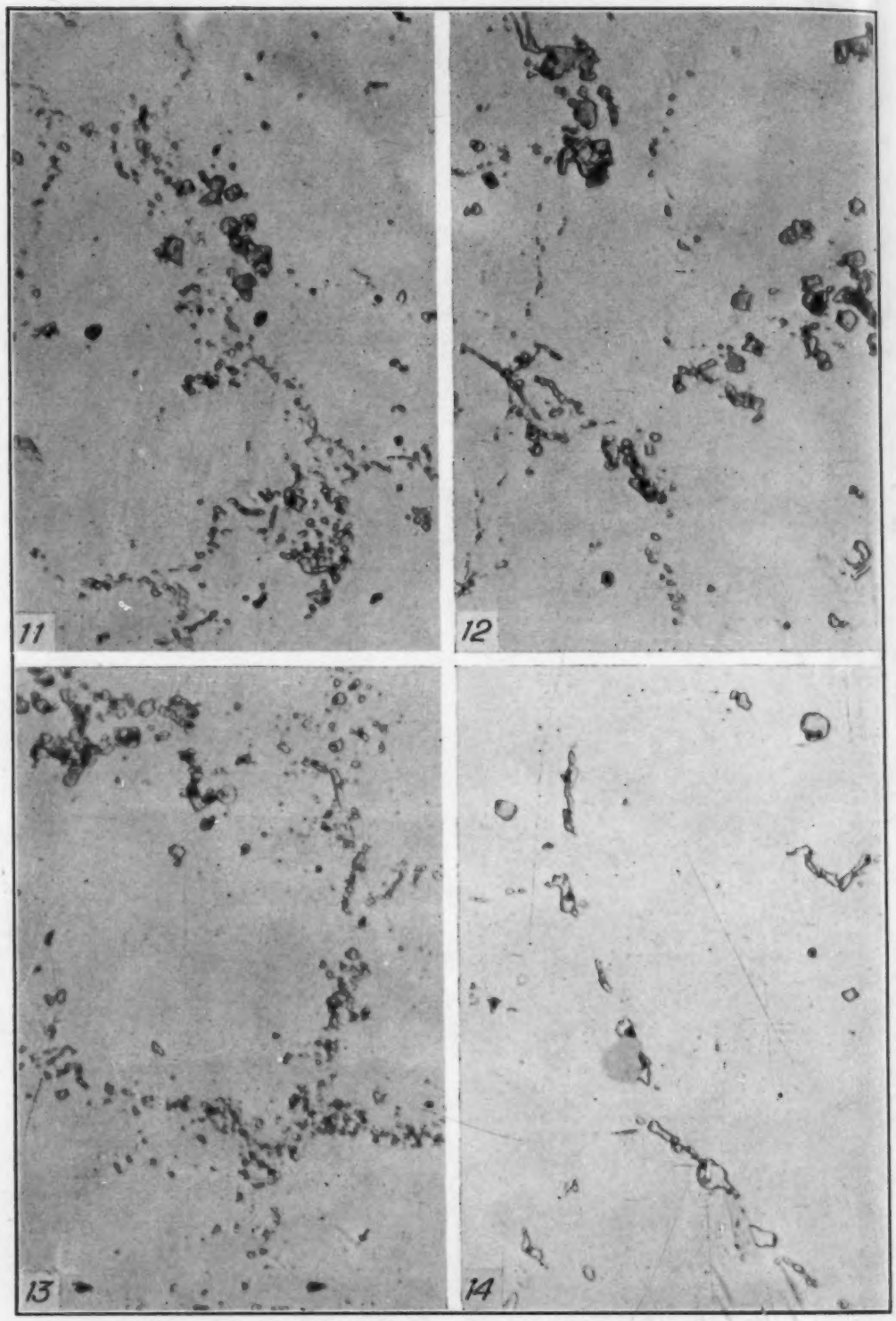
In order to check this, the samples were drawn for 8 hours more at 1650 degrees Fahr. (900 degrees Cent.), and furnace-cooled. The hardness values of these four are almost identical and are but little higher than those of the titanium-treated samples.

Becket and Franks[2] have mentioned the value of adding titanium to this type of alloy, because of its tendency to combine with the carbon present to form titanium carbide. This constituent being practically insoluble in the matrix, the resultant alloy can be considered as a carbon-free, high chromium iron. Referring to Fig. 6, the alloy D can be located at the extreme left end of the diagram (with complete insolubility of the carbide) since more than sufficient titanium is present to combine with all the carbon present. This mechanism

[2]Frederick M. Becket and Russell Franks,—American Institute of Mining and Metallurgical Engineers, Technical Publication No. 506, 1933.



Figs. 7 to 10—Samples of Alloy A Etched with Aqua Regia in Glycerine. × 100.
Fig. 7—Annealed 3 Hours at 1650 Degrees Fahr., Furnace-Cooled.
Fig. 8—Quenched from 2100 Degrees Fahr., Drawn 5 Hours at 1650 Degrees Fahr., Furnace-Cooled.
Fig. 9—Quenched from 2400 Degrees Fahr., Drawn 5 Hours at 1650 Degrees Fahr., Furnace-Cooled.
Fig. 10—Quenched from 2500 Degrees Fahr., Drawn 5 Hours at 1650 Degrees Fahr., Furnace-Cooled.



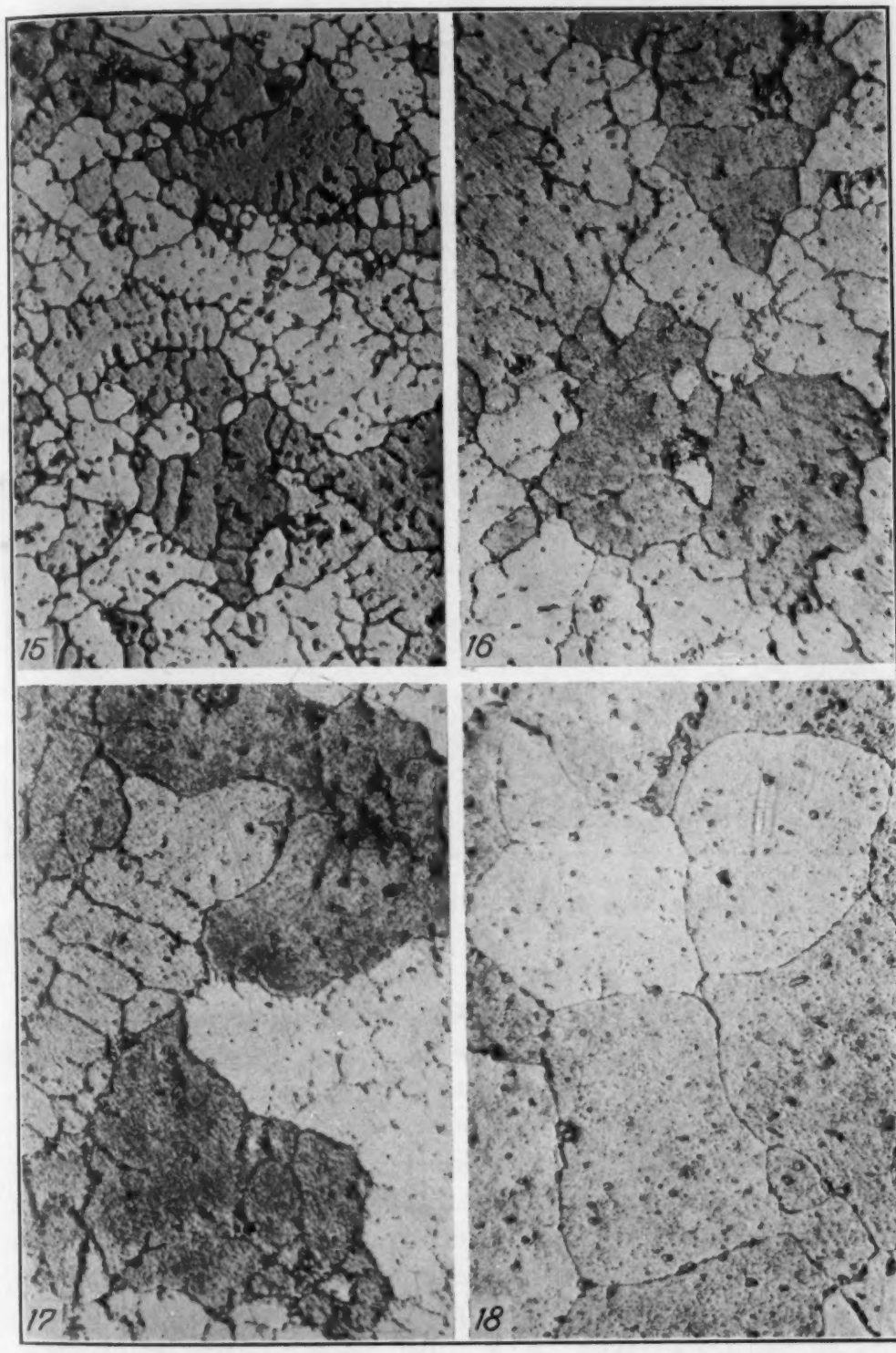
Figs. 11 to 14—Samples of Alloy D Unetched. × 500.

Fig. 11—As-Cast, Showing Crystals of the Titanium Constituent.

Fig. 12—Quenched from 2100 Degrees Fahr., Drawn 5 Hours at 1650 Degrees Fahr., Furnace-Cooled. Apparent Grain Size Same as Fig. 11.

Fig. 13—Quenched from 2250 Degrees Fahr., Drawn 5 Hours at 1650 Degrees Fahr., Furnace-Cooled. Apparent Grain Size Larger than Figs. 11 and 12.

Fig. 14—Quenched from 2500 Degrees Fahr., Drawn 5 Hours at 1650 Degrees Fahr., Furnace-Cooled. Apparent Grain Size Larger and Much Less Titanium Constituent in Evidence than Figs. 11, 12 and 13.



Figs. 15 to 18—Samples from Alloy D Etched with Aqua Regia in Glycerine. × 100.

Fig. 15—Sample As-Cast.

Fig. 16—Quenched from 2100 Degrees Fahr., Drawn 5 Hours at 1650 Degrees Fahr., Furnace-Cooled.

Fig. 17—Quenched from 2400 Degrees Fahr., Drawn 5 Hours at 1650 Degrees Fahr., Furnace-Cooled.

Fig. 18—Quenched from 2500 Degrees Fahr., Drawn 5 Hours at 1650 Degrees Fahr., Furnace-Cooled.

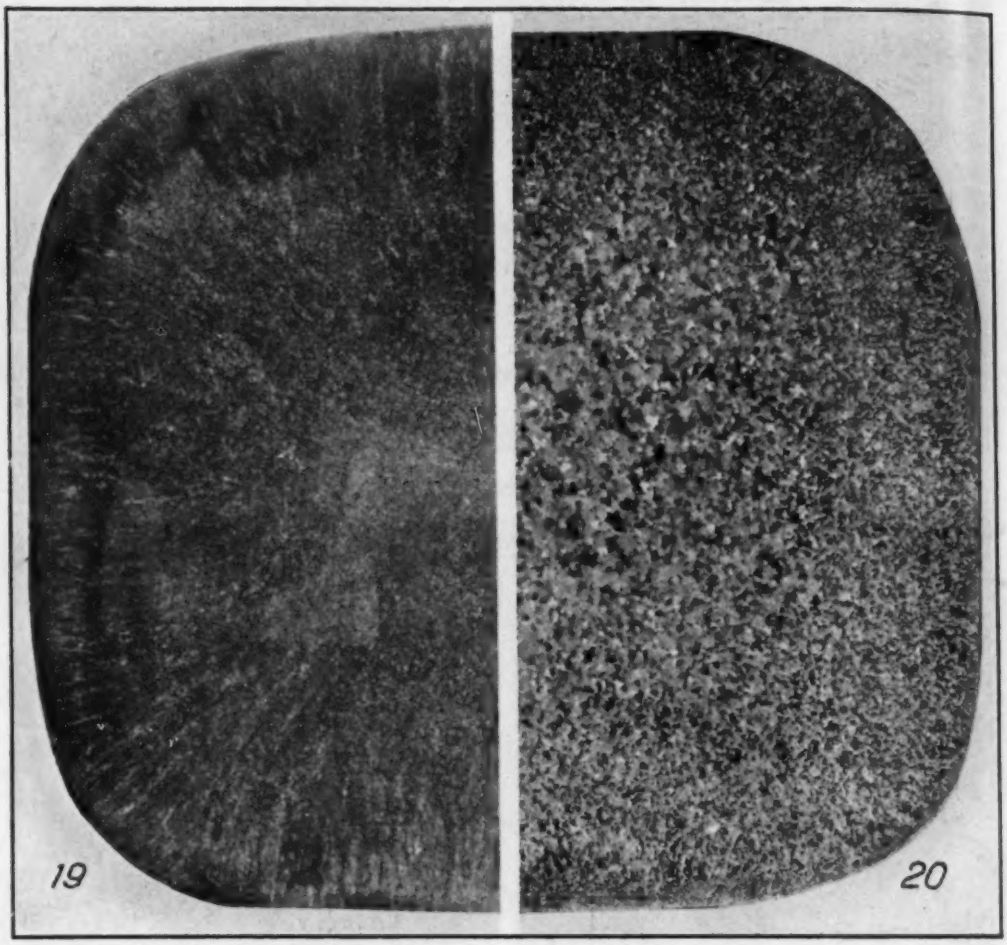


Fig. 19—Steel A, Etched with Chrome Regia. × 1.5.
Fig. 20—Steel C, Etched with Chrome Regia. × 1.5.

will result in complete lack of hardenability even in the water-quenched samples. This seems to be borne out by the hardness curves in Fig. 5 for alloy D. The only point at variance here is the hardness value of the sample quenched from 2100 degrees Fahr. (1150 degrees Cent.). A glance at the diagram in Fig. 6 shows that at this temperature the alloy is quite close to the left side of the gamma loop. Considering that a slightly lower chromium content below 18 per cent causes this loop to widen and considering also that there might be a slight solubility of titanium carbide plus the possibility of a very small amount of chromium carbide being retained, it is entirely possible that at this temperature a small amount of gamma was present which resulted in a very slight hardening when the alloy was quenched. The hardness values of the alloy as cast and of those quenched illustrate strikingly the absence of air-hardening and of

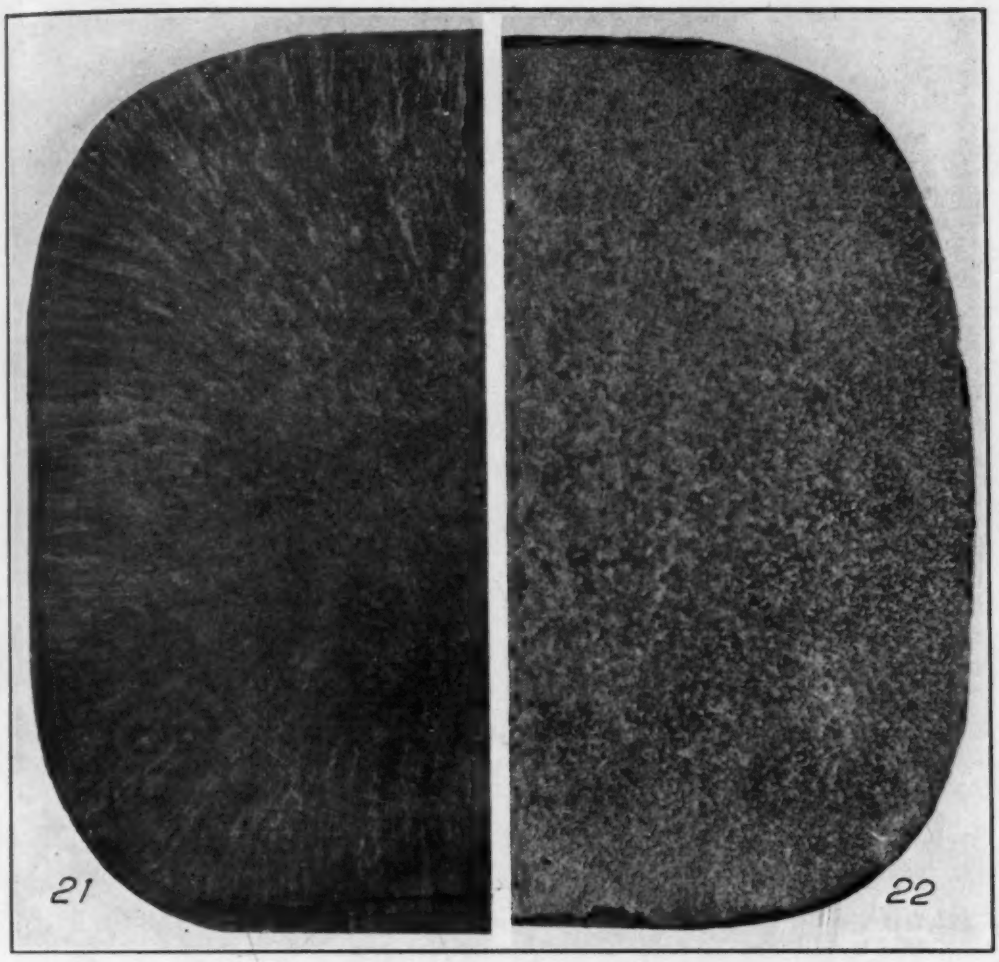


Fig. 21—Steel A, Etched with Sauveur's Reagent. × 1.5.
Fig. 22—Steel C, Etched with Sauveur's Reagent. × 1.5.

hardenability attendant upon the addition of titanium. This factor is of extreme importance in applications where machining is necessary on castings which it would be impractical to heat treat.

In the unetched samples from ingot D, shown in Figs. 11 to 14, differences in the distribution of the titanium constituent were noted. The crystals of this compound were quite plentiful and a large portion were segregated in a more or less "grain boundary" form. This apparent grain size was approximately the same in the sample quenched from 2100 degrees Fahr. (1150 degrees Cent.) and the sample as cast. There was no appreciable difference in any two samples quenched from the same temperature, one being subsequently drawn.

The apparent grain size as shown by the segregation of the titanium constituent was larger in the samples quenched from 2250

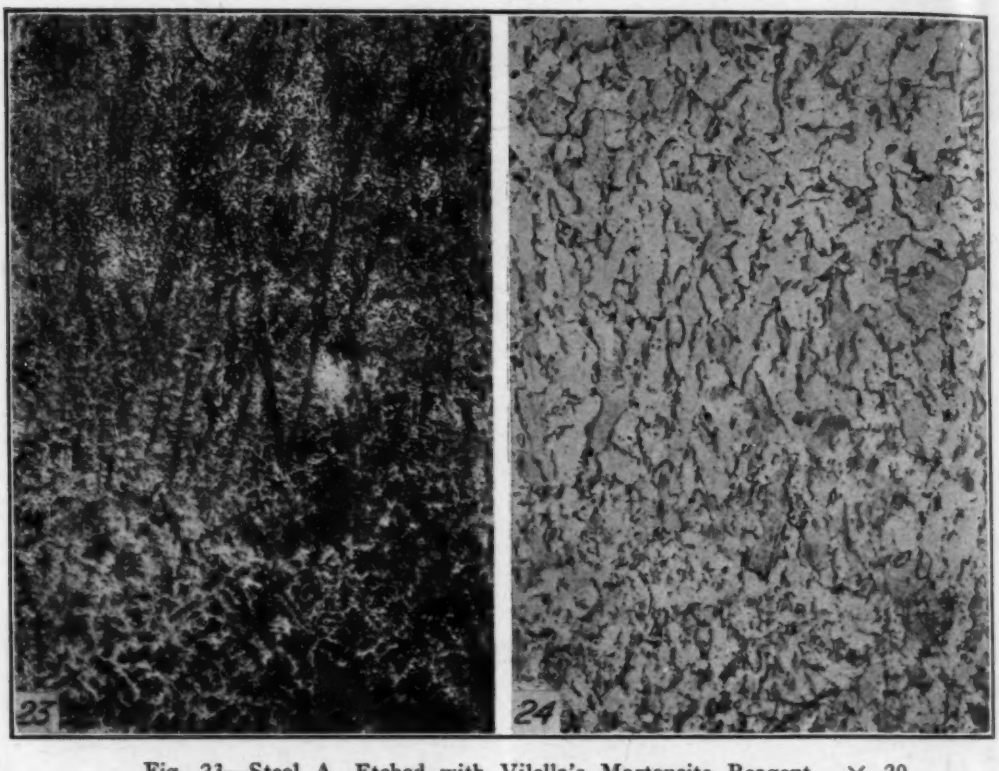


Fig. 23—Steel A, Etched with Vilella's Martensite Reagent. × 20.
Fig. 24—Steel C, Etched with Vilella's Martensite Reagent. × 20.

degrees Fahr. (1230 degrees Cent.). Those quenched from 2400 degrees Fahr. (1315 degrees Cent.) showed approximately the same structure.

The samples quenched from 2500 degrees Fahr. (1370 degrees Cent.), however, showed a marked difference in that there was appreciably less of the titanium constituent in evidence. In addition, the apparent grain size was considerably larger than in the samples treated at lower temperatures. This decrease in the amount of titanium constituent is in agreement with the results of E. Houdremont and P. Schafmeister,[3] who found that this constituent, which for convenience we shall refer to as titanium carbide, is soluble to some degree in austenite at 2460 degrees Fahr. (1350 degrees Cent.). Although in the present instance we are concerned with delta iron rather than austenite, the same condition seems to hold.

Since the microscopic evidence indicates that a portion of the titanium carbide went into solution at 2500 degrees Fahr. (1370 degrees Cent.) and no hardening resulted on quenching, it seemed possible that the alloy might be age-hardened by precipitation of this dis-

[3]E. Houdremont and P. Schafmeister, "Prevention of Corrosion of Grain Boundaries in 18 Per Cent Chromium—8 Per Cent Nickel Steels"—*Arch. Eisenhüttenwesen*, Vol. 7, p. 187-191, Sept. 1933.

solved constituent. Accordingly, samples which had been quenched from 2500 degrees Fahr. (1370 degrees Cent.) were drawn at temperatures of 1000, 1350, 1950 and 2200 degrees Fahr. (535, 730, 1065, 1205 degrees Cent.) for periods of time varying from 1 hour to 16 hours and air-cooled. There was however no appreciable increase in hardness, the Rockwell B values ranging from 83 to 86½.

Figs. 15 to 18 illustrate the microstructures of alloy D after etching. It was necessary to etch and re-polish a number of times in order to develop the structures of these samples. Considerable unavoidable pitting naturally resulted from this treatment.

The principal differences to be noted here are the increasing grain sizes with increased quenching temperatures and the disappearance of the dendritic structure in the sample heated to 2500 degrees Fahr. (1370 degrees Cent.). This can be accounted for by the solution of a large portion of the titanium carbide at this temperature. A good deal of this carbide had been present within the grains and served to outline the original dendritic formations.

Conclusions

1. The addition of titanium to 18 per cent chromium steel causes a refinement of grain in the ingot and a lessening of the tendency toward ingotism. With a titanium content of 1.11 per cent, ingotism is eliminated.

2. Although this alloy with sufficient titanium added to combine with all the carbon present may be considered for most practical purposes as a carbon-free, high chromium iron with titanium carbide inclusions, the crystals of the titanium compound are soluble in steel at 2500 degrees Fahr. (1370 degrees Cent.) and are therefore subject to re-arrangement by heat treatment; but the steel is not hardened by their solution.

3. The addition of 1.97 per cent titanium (or possibly less) to 18 per cent chromium steel with 0.20 to 0.25 per cent carbon eliminates both air-hardening and hardenability, except that water-quenching from the vicinity of 2100 degrees Fahr. (1150 degrees Cent.) may cause a very slight hardening.

Acknowledgments

The author wishes to express his appreciation to G. F. Comstock, metallurgist, in charge of metallurgical research, The Titanium Alloy Manufacturing Co., under whose direction this work was carried out.

Appreciation is also expressed to The Titanium Alloy Manufacturing Co. itself for permission to publish the results of this work.

DISCUSSION

Written Discussion: By V. N. Krivobok, Professor of Metallurgy, C.I.T., Pittsburgh, also Research Laboratory, Allegheny Steel Company, Brackenridge, Pa., and George C. Kiefer, Research Laboratory, Allegheny Steel Company, Brackenridge, Pa.

Technical literature of recent years already contains the information that additions of titanium to steels of the straight chromium stainless type renders these steels very much less hardenable by means of heat treatment. The work of Mr. Bannon, therefore, should be considered as a timely confirmation of the previous findings. Other information contained in this research paper should, perhaps, be taken up one by one.

Mr. Bannon believes that additions of titanium to 18 per cent chromium results in the marked refinement of grain in the ingot. This belief, if we judge correctly, is based upon the "deep etch" characteristics of ingots reproduced in Figs. 1, 2, 3, and 4. I should like to suggest that the reproduced appearance of the ingots is not altogether convincing. As Mr. Bannon states, these ingots have been subjected to different etching reagents. We know that under such circumstances it is difficult to judge the relative grain size of the ingots.

May we be permitted to add that this interesting observation has not been made before—to the best of our knowledge. Neither, in our opinion, are the submitted data altogether convincing. Of course, Mr. Bannon may have other experimental data to substantiate his observations and, if so, we would indeed be interested to hear it.

Further Beckett and Franks have pointed out to quote,[1] that "Titanium and columbium should be present in a ratio of 5 to 7 times the per cent of carbon. . . . . Large excesses of titanium and columbium over the recommended ratios bring about detrimental effects through hardening of the solid solution of iron and chromium."

As we judge from the paper, no tests except those just mentioned were made to determine whether or not the ingots in question really had the ingotism eliminated. Frankly we cannot find the justification for the statement, that, to quote, "With a titanium content of 1.11 per cent, ingotism is eliminated," as no additional experimental work has been made, or at least is not mentioned in the paper. It is rather well known that in steels with high concentrations of alloying elements a special technique is sometimes needed to bring out dendritic structure. In fact chromium is quite notorious in promoting segregation which is furthermore difficult to obliterate. Thus if the action of titanium is as it is said to be, it would be indeed important to know it.

The observation in regard to formation of a certain compound called by Mr. Bannon "titanium carbide" was of much interest to us, especially the observation that this constituent seems to be more soluble at high temperatures.

[1]F. M. Beckett and Russell Franks, *Transactions*, American Institute of Mining and Metallurgical Engineers, Vol. 113, p. 127-128.

Those who are concerned with fabrication of stainless steel containing titanium are likely to be familiar with the difficulties which arise from the presence of this constituent when its amount is as large as is shown in the paper (photo-

Figure Illustrating Mr. Sissener's Discussion

micrographs 11-14 inclusive) and its distribution seems to follow grain boundaries. Reduced impact characteristics may be expected.

The presence of such high concentrations of titanium may, and in our opinion will, affect corrosion resisting properties of these alloys. The presence of complete titanium bearing inclusions has a definite effect on corrosion properties of chromium-nickel stainless alloys and while we do not possess actual data on discussed alloys (because of high titanium content) it is logical to predict that the effect of titanium would be similar. Hence it may be suggested that even if titanium-bearing chromium alloys are benefited in certain respects, such as stated by Mr. Bannon, the conditions of service should be carefully considered before the use of the alloys is advocated.

Written Discussion: By J. Sissener, foundry manager, Myrens Verksted, Oslo.

I have with considerable interest read the work you have conducted in the field of the use of titanium in 18 per cent chromium iron, and published in the preprints of American Society for Metals. It ought to be of interest to you that we have conducted experiments on a large scale coming to the same conclusion as yourself in high chromium, high carbon castings.

As shown in the accompanying photograph (page 751), we use titanium in alloys with 27-30 per cent chromium and 0.7-1.2 per cent carbon. We generally find that an addition of below 0.05 per cent titanium is a remarkably good grain refiner when carbon is as high as 1 per cent. If, however, the titanium goes too far, as shown in the same photograph, above 0.05 per cent, we get several times the gas evolution, which gives porous castings. However, if the carbon is lower, the alloy is able to take up much more titanium. We cannot say how much, but evidently with lower carbon, we need much more titanium to refine the grain.

I find that small additions of titanium greatly improve the strength of high chromium cast irons.

Author's Reply

The discussion by Prof. Krivobok and Mr. Kiefer is much appreciated, and since they express doubt as to the value of the comparison between the macrostructures shown in the paper, further data on this point are gladly presented. Their chief criticism seems to be that the use of a different etching reagent on steels A and B invalidates the comparison obtained between the grain structures of these and the structures of steels C and D. Aqua regia in glycerine (used to develop the structures of C and D) had been tried on steels A and B and had shown the same pronounced ingotism shown in Figs. 1 and 2, but not as clearly. Indeed, the structures as developed with chrome regia were barely clear enough to photograph. Since the same structures were developed in A and B by the two reagents it was thought that the comparison was justified as stated in the paper.

However, in Figs. 19 and 20 are shown the structures of steels A and C, both etched with chrome regia. As can be seen here, the same comparison exists as regards the ingotism in steel A and the absence of it in steel C.

The question arises as to whether the reagents used had developed, in steels C and D, the structure of the individual grains and failed to show a dendritic formation of these that might have been present. For this reason a number of different reagents were tried on steels A and C and the results given herewith.

1. Neutral 10 per cent copper-ammonium chloride solution until the scratches were removed and then hydrochloric acid added until 25 c. c. had been added to 500 c.c. of solution. Used at 150-175 degrees Fahr. The same structure was obtained in steel C as had been obtained with aqua regia and chrome regia. Steel A was attacked vigorously in localized areas, spoiling the effect. The structure was never clear in this sample during the etching.

2. Hot (150-175 degrees Fahr.) ammonium persulphate solution.
   This developed, in steel C, just a faint indication of the granular structure already obtained with the reagents previously used. Not tried on steel A.
3. Chrome regia, to which copper-ammonium chloride was added in the ratio of 20 grams per 100 c.c. of solution. Used at 150-175 degrees Fahr.
   The same granular structure was obtained with steel C, perhaps a little clearer. The structure of steel A was the same as that obtained with chrome regia.
4. Hydrochloric acid 1:1. Used at 150-175 degrees Fahr. The structure of steel C was the same as already obtained. The structure of steel A was not as clear as that obtained with chrome regia.
   Further attempts were made to develop the structure of steel A more clearly, with the idea in mind to try the same reagent on steel C, which might tend to develop, if present, any heretofore hidden structure.
5. Ferric Chloride—30 gms.
   Hydrochloric acid—100 c.c.
   Copper Chloride—10 gms. Used at 150 degrees Fahr.
   Stannous Chloride—0.5 gms.
   Water—1000 c.c.
   The structure of steel A was not as clear as with chrome regia.
6. Water—3 parts Sauveur's Reagent
   Sulphuric acid—2 parts Used at 150 degrees Fahr.
   Hydrochloric acid—1 part
   The structure of steel A was somewhat more clear than with chrome regia. The addition of copper chloride to this reagent did not improve its action with respect to the clarity of the structure of steel A. The same was true regarding the addition of acetic acid to the reagent.

Figs. 21 and 22 show steels A and C after etching with this reagent.

Sections cut from the corners of ingots A and C were carefully polished and attempts made to develop the structures for examination at a somewhat higher magnification than was used in the work just described.

1. Ferric Chloride—5 gms.
   Hydrochloric acid—50 c.c.
   Water—100 c.c.
   This showed the dendritic structure of steel A to the eye but at 20 diameters magnification it would not have been discernible.
2. Ferric Chloride—10 gms.
   Hydrochloric acid—30 c.c.
   Water—q.s.—120 c.c.
   The results using this reagent were exactly the same as stated above.
3. Ethyl alcohol—95 c.c.
   Picric acid—1 gm. Vilella's Martensite Reagent.
   Hydrochloric acid—5 c.c.
   The structures developed by this reagent were sufficiently clear to photograph and are illustrated in Figs. 23 and 24.

Although the tests described here cannot be said to be exhaustive, the results would certainly seem to indicate a very strong probability that the conclusion stated in the paper is correct, viz., that a titanium content of 1.11 per cent eliminates ingotism in this type of steel. The possibility exists that the effect of this amount of titanium on other properties might be detrimental, but further work will have to be done to clear up this point and possibly "strike a balance" between the good and the bad effects of such additions.

Mr. Sissener's results as to the effect of titanium on the grain size of the higher chromium castings are of great interest, particularly so with reference to the small percentages of titanium used. The amounts shown are by no means enough to combine with all the carbon present. It would seem that for varying carbon ranges different amounts of titanium would be necessary to produce a desired effect. The chromium content may also have a distinct influence in this regard.

SOME EFFECTS OF SMALL ADDITIONS OF VANADIUM TO EUTECTOID STEEL

By J. G. Zimmerman, R. H. Aborn, and E. C. Bain

Abstract

This paper describes some of the effects of small additions of vanadium through a comparison of steels of approximately eutectoid composition, with and without vanadium. Observation of the transformation rate at a series of constant temperature levels, as affected by such factors as the austenitic grain size and the state of the carbides, affords definite evidence as to how small additions of vanadium affect the hardening properties of the steels.

THE purpose of this paper is to describe some of the effects of small vanadium additions through a comparison of steels of approximately eutectoid composition, with and without vanadium. The influence of vanadium in steel has been studied by several investigators, generally, however, with regard to its effect on the physical and mechanical properties; the problem is here approached through an investigation of the transformation and tempering characteristics of the steels in question, and the fundamental contribution of vanadium is recognized from its effect upon these two characteristics.

One means of comparison is afforded by study of the constant temperature transformation of austenite to ferrite and carbide, a reaction which tends to occur whenever a steel, previously heated to form a solid solution of the carbon in gamma iron (austenite), is rapidly cooled to a temperature below that at which the austenite is really stable. Fortunately, for purposes of study as well as for practical uses, this reaction does not immediately get under way as soon as the temperature of the austenite is suitably depressed; therefore by quenching into a sufficiently effective medium the progress and result of this reaction at any constant temperature may be studied by any means which differentiates the unchanged austenite from its transformation products. This has been the subject of more extensive discussion (1)[1], and it is here only mentioned as affording a satis-

[1]The figures appearing in parentheses pertain to the references appended to this paper.

A paper presented before the Eighteenth Annual Convention of the Society held in Cleveland, October 19 to 23, 1936. When the experimental work described in this paper was done, all of the authors were members of the staff of the Research Laboratory, United States Steel Corp., Kearny, N. J. Manuscript received May 18, 1936.

factory basis for studying the fundamental contribution of vanadium.

There is no longer any doubt that the final properties of a carbon or low alloy steel of high carbon content, which can be so widely varied by heat treatment and processing, are mainly determined by the microstructure of the product, rather than by the mere presence of an element in one or more of the constituents which make up the structure. Obviously, then, an alloying element may act to enhance or improve these properties most strongly if it profoundly influences, by its presence, the structures which may be developed. In so doing, the element may be included in the final useful structure in only three possible ways:

1. in solid solution in the ferrite,
2. in the carbide,
3. in special intermetallic compounds, oxides, etc.

This simple statement is possible because very few steels as ready for service contain other than the two constituents ferrite and carbide, in one or more of their various associations. Austenite and untempered martensite are very rarely present in steels as used, and are not here considered. The elements may be classified on the basis of their occurrence in the steel as outlined above (1, 2 or 3). Typical representatives of the first class are nickel, cobalt, silicon, aluminum, and to some extent manganese; they go chiefly into solid solution in the ferrite phase, there to strengthen the matrix.

Only brief mention need be made here of the third class. Intermetallic compounds in steels are believed to be exceptional, and there is no evidence that vanadium when present in such small amounts as herein discussed forms any such compound with iron. It does, of course, form such nonmetallic compounds as oxides (vanadia) and possibly others, but as it is seldom added to any except thoroughly deoxidized steels, such as used in this investigation, these compounds need not be considered at length.

Evidence will be presented to show that vanadium as customarily used falls into the second class along with elements such as molybdenum, tungsten, titanium, and chromium, which are preferentially carbide-forming, although definitely ferrite-soluble in themselves when present in amounts in excess of that necessary to fulfill the requirements of the carbon. Vanadium is believed to play an interesting and important rôle even when present in such relatively small proportions as are discussed here.

Investigations of vanadium steel by Arnold and Read (2) led

them to believe that the carbide of vanadium was V_4C_3*, and they showed that if vanadium were present in sufficient quantity its carbide completely replaces the iron carbide, Fe_3C, although the exact form of the carbon solubility line has not yet been determined. Abram (3), in a recent paper, investigated the influence of vanadium additions to alloy steels and very low carbon steels, particularly with reference to their behavior on tempering. Houdremont, Bennek, and Schrader (4) have made extensive studies on the hardening and tempering of steels of the type here discussed as affected by additions such as 1, 3 and 6 per cent vanadium; this particularly valuable paper gives a clear insight into the major effects of vanadium when present in higher concentrations.

The observations presented here show how even relatively small amounts of vanadium definitely affect the behavior of high carbon steels. Transformation rates and the effect upon them of such factors as grain size and carbide behavior, both influenced, in turn, by vanadium, constitute the main basis of study; certain additional experiments along other lines provide explanation for minor contributions of vanadium.

Material Used

Four steels, all closely approximating eutectoid composition, were used. The hardenability and tempering tests were made on 1-inch diameter hot-rolled bar stock; all other tests were made on 1/16-inch cold-rolled strip. Steels C1 and V1 were from experimental heats, while C2 and V2 were selected from commercial tool steel heats. The analyses follow:

	C1	C2	V1	V2
Carbon	0.88	0.89	0.90	0.87
Manganese	0.41	0.29	0.47	0.30
Silicon	0.41	0.15	0.42	0.32
Vanadium	...	...	0.20	0.27

Influence of Austenitic Grain Size

It was an early observation, and now a familiar one, that the fracture of a vanadium-bearing steel, which had been quenched from the higher temperature range for plain carbon steels, usually exhibits a very fine texture as compared to that of a carbon steel similarly

*A controversial point. Considering the method of determination, it may well be that the carbide is VC.

treated. Such steels were promptly designated as fine-grained, according to the common criterion of textures of fractures, and correctly so, for, generally speaking, the texture of a martensitic fracture correlates well with austenitic grain size. It was likewise observed that for treatments at progressively higher temperature the fracture of the vanadium steel becomes more and more like that of the plain carbon steel heat treated at somewhat lower temperatures; or, in effect, that the austenite of vanadium steel also goes through a process of grain coarsening, but only at a higher temperature than for carbon steel. Thus, through its influence on grain size, vanadium in steel was known to widen effectively the heat treating range, a fact contributing to its early use.

Since the austenitic grain size of a steel is an important factor in its response to heat treatment, it was desirable to make a more precise investigation of the grain growth characteristics for both the carbon and vanadium steels by actually determining the exact austenitic grain size developed at various temperatures. This was carried out on each of the steels studied, and at the same time a qualitative measure of the extent of carbide solution was made. In Table I is given a typical series of the quenching temperatures employed, and for each temperature the corresponding grain size developed and the degree of carbide solution.

Table I

Quenching Temperature		Grain Size (A.S.T.M.)**		State of Carbides	
Fahrenheit	Centigrade	Carbon Steel	Vanadium Steel	Carbon Steel	Vanadium Steel*
1400	760	6	10-11	Partially dissolved	Undissolved
1500	815	6	9-10	Essentially dissolved	Undissolved
1600	870	5	8	Essentially dissolved	Undissolved
1700	925	4	7	Essentially dissolved	Partially dissolved
1800	980	3-4	6-7	Essentially dissolved	Partially dissolved
1900	1040	3-4	3-4	Essentially dissolved	Largely dissolved
2000	1095	2-3	2-3	Essentially dissolved	Essentially dissolved
2100	1150	2-3	0	Essentially dissolved	Essentially dissolved

*Represents carbide solubility in 0.27 per cent vanadium steel. Similar carbide behavior noted in 0.20 per cent vanadium steel, but at somewhat lower temperature.

**Extension beyond Nos. 1 and 8 determined by method described by Davenport and Bain, TRANSACTIONS, American Society for Metals, Vol. 22, 1934, p. 879.

Since the compositions of the steels were such that little or no proeutectoid constituent could be rejected to outline the grain

boundaries of the austenite on cooling through the critical range, it was necessary to devise another means for estimating the grain size. This was accomplished by only partly immersing the specimen, previously rendered austenitic, in the quenching medium. As a result one end was successfully quenched to form martensite while the other end, exposed only to air, transformed wholly to pearlite. In each case there was an intermediate zone which in effect was cooled at a rate just less than the limiting quenching rate necessary to produce martensite; and owing to the fact that the transformation to fine pearlite usually begins at the grain boundaries, microscopic examination clearly revealed where the grain boundaries of the parent austenite were located.

The vanadium effect previously referred to can be readily observed in Table I; that is, up to about 1800 degrees Fahr. (980 degrees Cent.) the vanadium steel is distinctly fine-grained, whereas the grain size of the carbon steel at that temperature is definitely coarse. Above 1800 degrees Fahr. (980 degrees Cent.) however, the vanadium steel coarsens abruptly, for it then enters its elevated coarsening range. The effective widening of the heat treating range now becomes apparent, for by comparing the two steels from the standpoint of the temperature required to produce in each a coarse grain size, for example No. 4, it is seen that this grain size is developed in the carbon steel at 1700 degrees Fahr. (925 degrees Cent.) whereas not until 1900 degrees Fahr. (1040 degrees Cent.) does the vanadium steel develop a comparable grain size.

The question now arises as to just how vanadium inhibits grain growth. Possible clues are afforded by reference to Table I and also by Fig. 1a and 1b, a comparison of micrographs of steels C2 and V2 as quenched into brine from 1650 degrees Fahr. (900 degrees Cent.). As is easily seen, the vanadium steel is showered with an exceedingly large number of carbide particles, quite certainly a vanadium-rich carbide, undissolved in the heating and scattered at random throughout the specimen, whereas the iron carbide in the plain carbon steel is essentially completely dissolved. This shows that vanadium-rich carbides are much less soluble than iron carbides, and consequently that the particles persist as such to a higher temperature in the steel. Such particles doubtless promote simultaneous transformation on many nuclei as the steel cools through the appropriate temperature range; but, what is more important, the carbide particles appear to act not only as nuclei but also as grain growth inhibitors, perhaps by

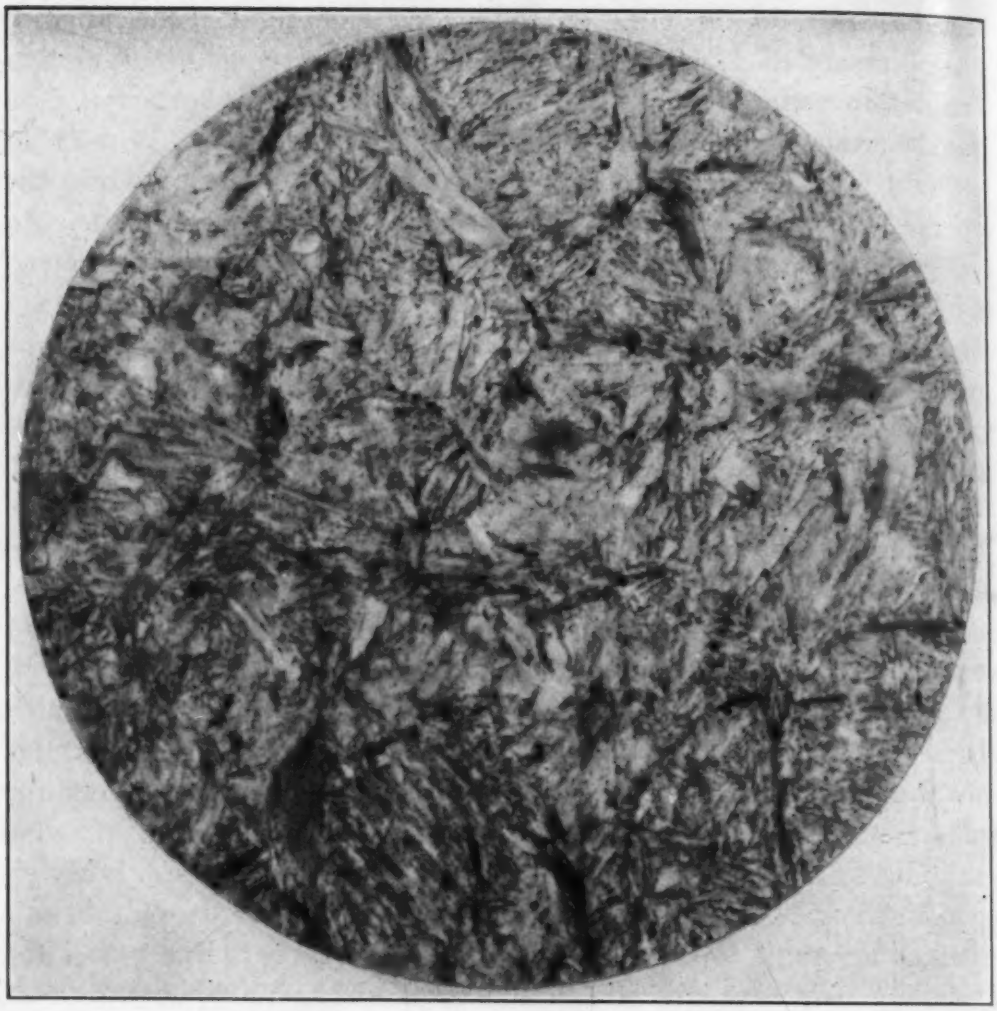


Fig. 1a and 1b—Difference in Carbide Solubility at 1650 Degrees Fahr. Between Carbon Steel C2 and Vanadium Steel V2. × 1000. Fig. 1a—Carbon Steel C2, Quenched from 1650 Degrees Fahr. (cf. Fig. 1b).

increasing the number and uniformity of the austenite grains formed upon heating. The temperature of marked grain growth is thus raised to a much higher level (because of the inhibiting effect of these undissolved carbides) and so the temperature range is raised to which the steel may be heated without undue grain-coarsening. Thus the shortcomings (such as brittleness) of coarsened plain carbon steel are minimized.

An important point to be remembered in this connection is the fact that vanadium imparts this action only when present as the undissolved carbide. The steel may properly enough be considered as reluctant to coarsen, since at ordinary heat treating temperatures it remains fine-grained; nevertheless if the temperature is raised to a

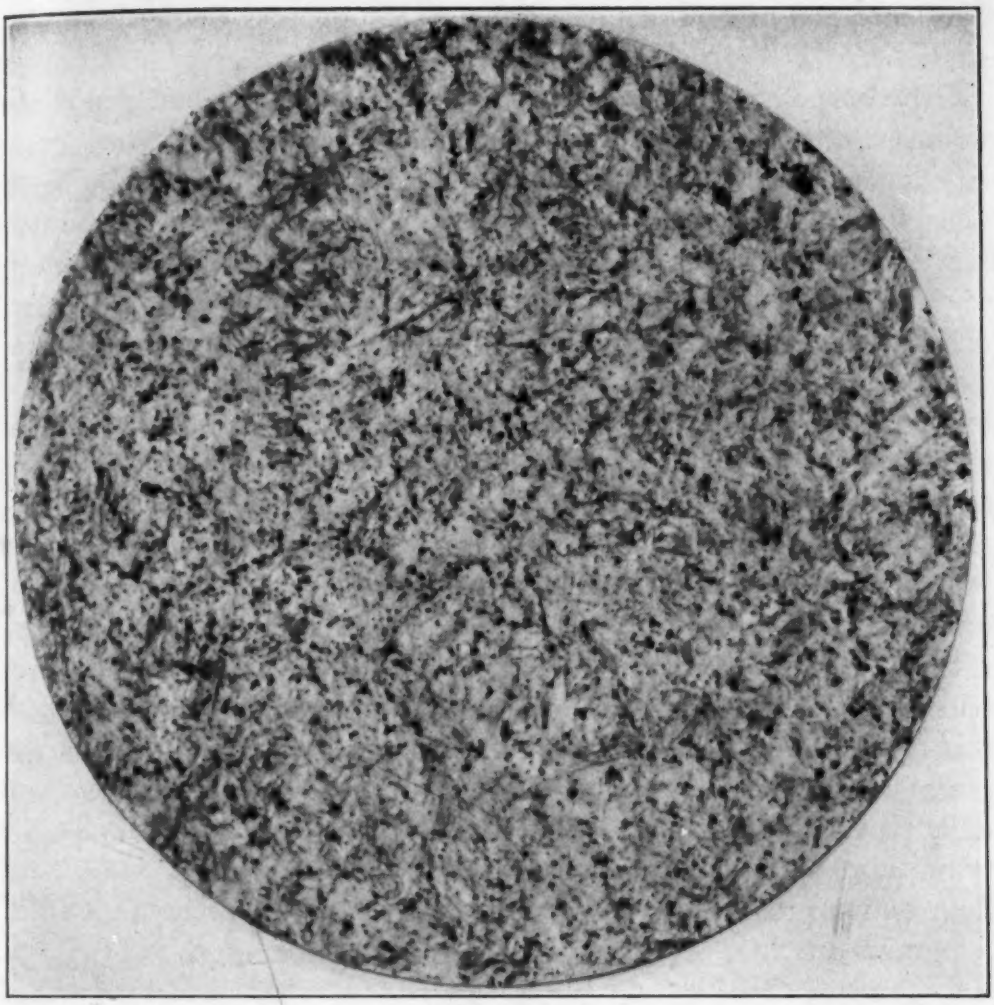


Fig. 1b—Vanadium Steel V2 Quenched from 1650 Degrees Fahr. (cf. Fig. 1a).

point permitting complete solution of the vanadium carbide particles, the inhibition is clearly removed and the steel then enters a temperature range of marked grain growth (which the carbon steel had already attained at a much lower temperature). When the particles are all dissolved the vanadium steel may, in fact, develop a grain size equal to, or even surpassing, that developed in the plain carbon steel at the same temperature. Indeed, in most commercial steels, the finer the initial grain established the higher the temperature at which marked growth occurs, but the coarser will be the grains finally developed. Referring again to Table I, the coarsening of the vanadium steel above 1800 degrees Fahr. (980 degrees Cent.) is observed to coincide with solution of the vanadium carbides, following which the vanadium steel rapidly approaches the carbon steel in grain size

developed and at 2100 degrees Fahr. (1150 degrees Cent.) even surpasses it.

Having in mind the mechanism of grain establishment and the influence of vanadium upon it, attention now turns to just how grain size alone is one of the dominant factors determining the rate at which reactions proceed in the solid state. It was early observed that a fine austenitic grain size or fine fracture is a usual accompaniment of the development of soft spots in certain carbon steels during an ordinary hardening treatment. It now appears that the fine grain size in itself is in large part responsible for this behavior in that it brings about a more rapid transformation of austenite to ferrite and carbide (5); or, conversely, by heating a steel to successively higher temperatures and thus establishing a larger and larger grain size, the rate of transformation on cooling is progressively retarded. That this is indeed so can be readily observed by reference to Fig. 4 (a), a time-temperature curve showing the rate of the austenite transformation at a series of constant subcritical temperatures. Such curves will be taken up in detail later, but it is of importance to note here that as the heating temperature is raised, in consequence of which the grain size progressively increases, the curve is displaced correspondingly toward the right, that is, toward an increased time interval. To summarize, therefore, the influence of grain size in itself is to retard the reaction rate when the grains are coarse and to accelerate it when the grains are fine.

Hardenability

"The hardenability of a steel is a function of its rate of transformation, in the early stages, at a temperature at which this rate is a maximum" (5).

There is, in the transformation of austenite to ferrite and carbide, a temperature region (around 1000 degrees Fahr. (540 degrees Cent.) for carbon steels) where the reaction is most rapid, and it is the transformation at this temperature which is to be obviated in a successful quench. The hardenability of a steel (in the quenching of a given section) is measured by the depth below the surface at which the austenite cools at a rate sufficient to prevent this rapid reaction. Obviously, then, any factors tending either to retard or to accelerate this maximum rate may be considered as controlling the hardenability of a steel, and in inquiring into the influence of vanadium in this respect it is only necessary to refer to certain details

already discussed, for the transformation rate at any given temperature is in itself determined by two factors: effective grain size, and the composition of austenite phase with regard to dissolved elements.

A steel as quenched from higher and higher temperatures into water or brine generally becomes successively more and more deep-hardening, a fact which has been shown by other investigations to be a function of the effective grain size developed by increasing temperature (5). The retarding effect of coarse grains on the rate of transformation has been pointed out above, and it can readily be inferred that the net effect of grain coarsening on hardenability is to increase, in the temperature region of most rapid reaction, the time interval required before this transformation starts, thus permitting deeper zones in the metal to become martensitic.

Opposing this tendency, vanadium, as long as it is present as the undissolved carbide, retards grain growth and consequently induces shallow hardening because, as has been shown, fine grains accelerate the austenite transformation. In addition, the carbide particles in themselves further promote shallow hardening by acting as nuclei to initiate transformation, (thereby having the effect of an even finer grain size); and because of their random dispersion the transformation is not restricted primarily to grain boundaries, but may begin at many points, even within the body of the individual grains.

With reference to the second factor controlling the hardenability, that of austenite composition with regard to dissolved elements, it now appears that any element thus dissolved (with the possible exception of cobalt) contributes to deep-hardening. Typical examples in this respect are afforded in the familiar effect of such elements as chromium, nickel, and manganese, which operate, when dissolved in the austenite, to make it very sluggish in starting to transform and thus promote deep-hardening. When the temperature is sufficiently high to dissolve the carbides in a vanadium-bearing steel, this element acts in much the same manner. The composition of the austenite is altered by the vanadium in solution, and its transformation is correspondingly retarded. Thus, paradoxically, vanadium can be both deep- and shallow-hardening in its effect. When present as the undissolved carbide it promotes shallow-hardening, but when dissolved in the austenite its effect is toward deep-hardening.

That this is indeed the case is exemplified by reference to Fig. 2a and 2b which compares the plots of hardness values taken across the diameters of two of the steels, one with and one without vanadium,

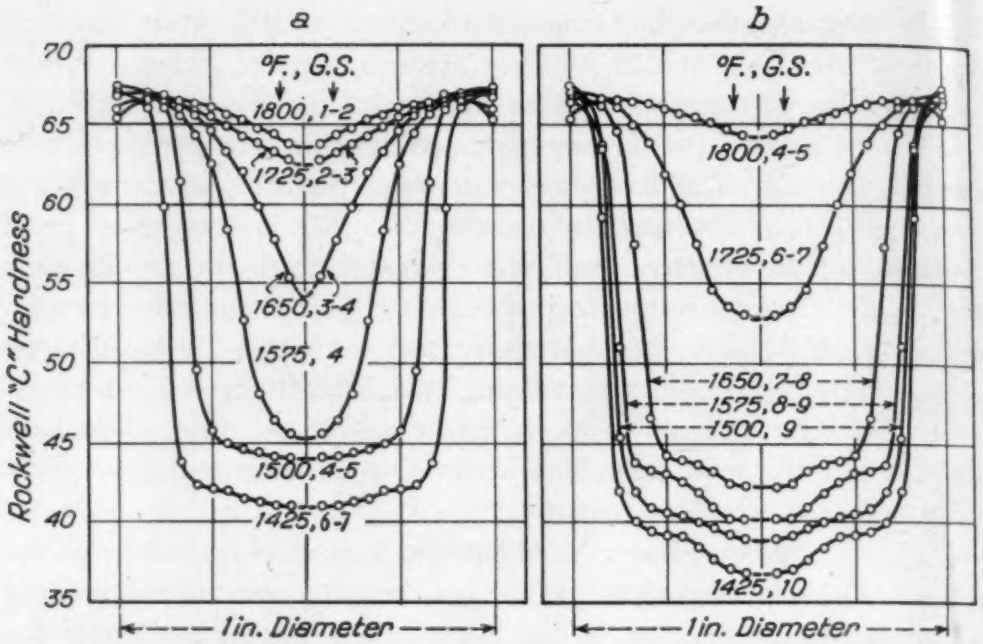


Fig. 2a—Hardenability of Carbon Steel C2 as Affected by Successively Higher Quenching Temperature.

Fig. 2b—Hardenability of Vanadium Steel V2 as Affected by Successively Higher Quenching Temperature.

similarly heated and quenched from successively higher temperatures into brine. The hardenability of the plain carbon steel can be observed to increase progressively with grain coarsening, while in the vanadium steel of practically the same composition there is no appreciable increase in the depth of hardening until a temperature (e.g. 1725 degrees Fahr.) (940 degrees Cent.) is reached at which the vanadium-rich carbide begins to dissolve in substantial proportions; after which, for comparable heating temperatures, it soon equals and then surpasses the depth of hardening of the plain carbon steel. An interesting comparison of the respective hardenabilities is afforded by considering the depth of hardening in the two steels for a similar grain size, particularly for the grain-size number 4-5. At this grain size the vanadium steel is even slightly deeper hardening than the deepest of the carbon series, and is far deeper hardening than the carbon steel with the same grain size because of the sluggishness imposed by the dissolved vanadium.

Fig. 2 also illustrates an important point typical of the apparently complex influence of vanadium. In the curve of the hardness plots for the 1725 degrees Fahr. (940 degrees Cent.) quench it will be seen that although the vanadium steel is as yet comparatively fine-grained (No. 6-7), it nevertheless is moderately deep-hardening,

certainly much more so than the carbon steel having the same grain size. Here then is a fine-grained steel exhibiting moderately deep-hardening effects, which is an apparent contradiction; but in reality it is the summation of two opposing tendencies; it is the result of partial carbide solution, in consequence of which the effective alloy content of the austenite is increased, thus promoting deep-hardening as explained above, while at the same time there are enough remaining carbide particles to restrict grain growth and retain a fine-grained structure.

Transformation Rates

So far little has been said, in other than a general way, of the actual differences in the rate of austenite transformation as brought about by vanadium addition; attention is now directed to some phases of this behavior at a series of temperatures below Ae_1, the transformation temperature at zero rate of heating or cooling. Fundamentally, the study is that of the time interval required for austenite, brought rapidly from one of a series of temperatures above Ae_1, to transform to ferrite and carbide at one of a series of temperature levels below Ae_1.

In order to observe the progress and result of austenite decomposition at any constant temperature it is of course necessary to cool the properly heated specimen rapidly to the chosen bath temperature and then to maintain it at that temperature within narrow limits. The quenching action must be rapid enough to preserve the austenite essentially unchanged down to the bath temperature so that the material will be given no choice but to transform at that precise constant temperature. In practice this was accomplished by the use of small, thin specimens and of a quenching bath of high cooling power, molten lead (or lead-bismuth at temperatures below the practical limits for lead), controlled automatically at the level to be studied. For those temperature levels ranging from just under the lower critical down to about 400 degrees Fahr. (205 degrees Cent.) the progress of the transformation may be followed by a method of high reliability. The procedure consists in quenching a series of specimens (from a temperature in the stable austenite zone) directly into a molten bath at the desired subcritical level, holding each for a definite time interval, and then quenching it quickly into brine at room temperature. In this manner any austenite which has not transformed to ferrite and carbide at the constant temperature level is transformed to marten-

site in the brine quench, and subsequent microscopic investigation reveals in sharp contrast the progressive loss in austenite and gain in transformation products as specimens are held in the intermediate temperature bath for successively longer time intervals. Those specimens removed from the lead bath prior to the onset of any transformation develop full martensitic hardness (Rockwell C 65-66), whereas those removed to the brine after considerable transforma-

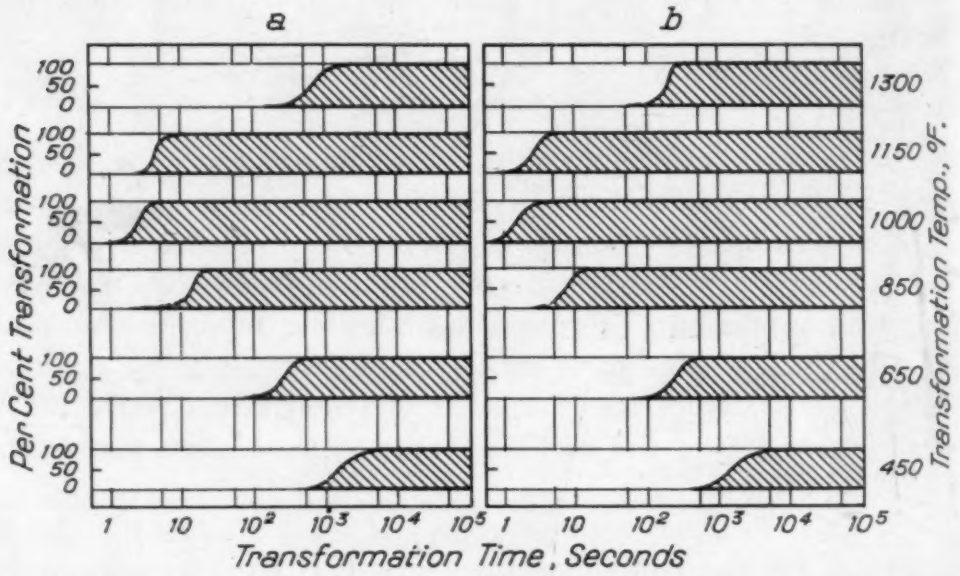


Fig. 3a.—Transformation Rates in Carbon Steel C2 Transferred Directly from 1500 Degrees Fahr. to Indicated Temperature Levels. Austenitic Grain Size 5-6.

Fig. 3b—Transformation Rates in Vanadium Steel V2 Transferred Directly from 1500 Degrees Fahr. to Indicated Temperature Levels. Austenitic Grain Size 11.

tion has occurred are increasingly soft, reaching finally the hardness of the fully transformed product characteristic of that particular temperature level. Thus both hardness and structure trace the progress of austenite transformation.

This procedure was followed at all transformation levels studied, and the direct evidence from microscopic examination was recorded in terms of per cent austenite unchanged or of transformation products already formed. A tabulated summary may be found in Table II, while typical data are presented graphically in two forms:

1. Fig. 3 presents two examples of the plot of time interval versus the percentage transformed at each of the several levels, for a typical heating temperature as employed for steels C2 and V2. At any given time the proportion of the remaining unchanged austenite is represented by the width of the white band; that of the transformation products by the width of the shaded band.

Table II

Time-Intervals for Transformation of the Carbon and Vanadium Steels at a Series of Constant-Temperature Levels

Transf. Level °F.	Heating Temp. °F.	Resulting Aust. Grain Size		Transformation of C Steel			Steel C2 (Seconds)		Transformation of V Steel					
				Steel C1 (Seconds)					Steel V1 (Seconds)			Steel V2 (Seconds)		
		C Steel	V Steel	Begins	50%	Ends	Begins	50%	Ends	Begins	50%	Ends	Begins	50%	Ends
1300	1500	6	11	____	____	____	150	450	1500	____	____	____	55	127	185
1300	1550	5	10-11	150	500	1400	____	____	____	50	160	240	____	____	____
1300	1625	4-5	____	____	____	____	350	1320	4000	____	____	____	____	____	____
1300	1650	4-5	7-8	250	1150	2500	____	____	____	150	245	400	____	____	____
1300	1850	2-3	4-5	____	____	____	900	4400	7500	150	410	750	200	440	800
1300	1925	____	2-3	____	____	____	____	____	____	____	____	____	150	490	800
1300	2000	2-3	2-3	1000	3300	7000	____	____	____	100	360	510	____	____	____
1150	1500	6	11	____	____	____	2	4	10	____	____	____	<1	2	4
1150	1550	5	10-11	<1	4	5	____	____	____	1	3	6	____	____	____
1150	1625	4-5	____	____	____	____	2	5	8	____	____	____	____	____	____
1150	1650	4-5	7-8	1	3	5	____	____	____	2	4	11	____	____	____
1150	1850	2-3	4-5	____	____	____	2	9	12	4	10	18	5	11	15
1150	1925	____	2-3	____	____	____	____	____	____	____	____	____	5	17	30
1150	2000	2-3	2-3	4	11	17	____	____	____	4	14	25	____	____	____
1000	1500	6	11	____	____	____	<1	2	4	____	____	____	<1	1	3
1000	1550	5	10-11	<1	3	6	____	____	____	<1	2	5	____	____	____
1000	1625	4-5	____	____	____	____	1	2	5	____	____	____	____	____	____
1000	1650	4-5	7-8	1	3	6	____	____	____	2	4	10	____	____	____
1000	1850	2-3	4-5	____	____	____	2	5	8	5	16	30	5	18	50
1000	1925	____	2-3	____	____	____	____	____	____	____	____	____	6	24	50
1000	2000	2-3	2-3	3	8	10	____	____	____	5	20	35	____	____	____
850	1500	6	11	____	____	____	1	12	20	____	____	____	1	6	8
850	1550	5	10-11	____	____	____	____	____	____	____	____	____	____	____	____
850	1625	4-5	____	____	____	____	2	12	30	____	____	____	____	____	____
850	1650	4-5	7-8	1	21	40	____	____	____	2	14	28	____	____	____
850	1850	2-3	4-5	____	____	____	3	21	45	5	44	80	6	26	60
850	1925	____	2-3	____	____	____	____	____	____	____	____	____	10	43	100
850	2000	2-3	2-3	3	22	40	____	____	____	10	56	95	____	____	____
650	1500	6	11	____	____	____	40	235	500	____	____	____	30	210	400
650	1550	5	10-11	____	____	____	____	____	____	____	____	____	____	____	____
650	1625	4-5	____	____	____	____	50	265	500	____	____	____	____	____	____
650	1650	4-5	7-8	60	340	600	____	____	____	40	315	800	____	____	____
650	1850	2-3	4-5	____	____	____	70	310	600	80	400	800	50	285	700
650	1925	____	2-3	____	____	____	____	____	____	____	____	____	70	340	800
650	2000	2-3	2-3	50	265	600	____	____	____	100	540	1000	____	____	____
450	1500	6	11	____	____	____	360	1200	5400	____	____	____	270	1080	6000
450	1550	5	10-11	____	____	____	____	____	____	____	____	____	____	____	____
450	1625	4-5	____	____	____	____	300	1650	6000	____	____	____	____	____	____
450	1650	4-5	7-8	840	2820	15000	____	____	____	840	2820	15000	____	____	____
450	1850	2-3	4-5	____	____	____	360	1710	6600	720	1980	15000	410	1480	6500
450	1925	____	2-3	____	____	____	____	____	____	____	____	____	240	1360	9000
450	2000	2-3	2-3	300	1800	21000	____	____	____	300	2400	14400	____	____	____

NOTE: Grain size numbers on expanded A.S.T.M. scale. Trace or <0.1% of transformed product or austenite represents beginning and end point respectively.

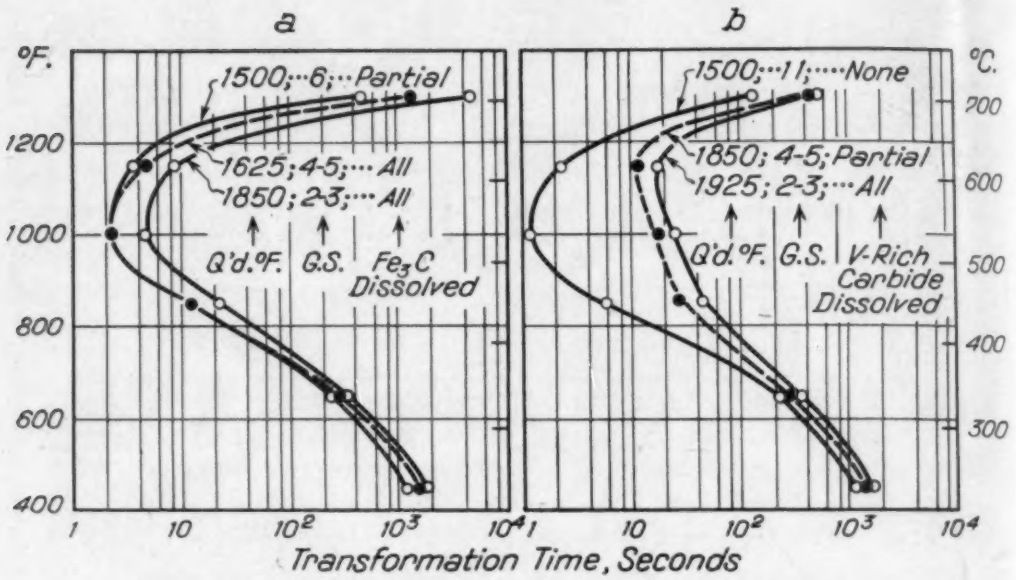


Fig. 4a—Comparative Time Intervals for 50 Per Cent Transformation in Carbon Steel C2 as Quenched from Successively Higher Temperatures. Note Fixed Temperature of "Nose".

Fig. 4b—Comparative Time Intervals for 50 Per Cent Transformation in Vanadium Steel V2 as Quenched from Successively Higher Temperatures. Note Rise of "Nose" with Quenching Temperature.

2. Figs. 4 to 6 present, in each case, the time required for the transformation to proceed half way to completion at each of the subcritical levels. In nearly all cases the relative position of the curves for the vanadium and for the carbon steel proved to be the same whether the basis of comparison was the beginning, the half-way period, or the end of the transformation. The 50 per cent points were chosen as representative of the rate at which the reaction proceeds at that level; upon close examination it will be seen that the course of these curves can be traced by connecting the mid-points of the corresponding curves in Fig. 3.

In both types of plots, the abscissa is the time interval, on a logarithmic scale necessitated by the wide range in the rate for different transformation temperatures. The complete transformation at a level of slow reaction may require a time interval over 100,000 times as long as at a level of rapid reaction; therefore detail in the short time intervals could not be shown practically on a linear scale. The complete development and analysis of such curves has been described in detail in an earlier and more extensive paper (1); a brief survey of the method has been presented here only as an outline of the procedure employed and as an aid to the reader unfamiliar with this technique.

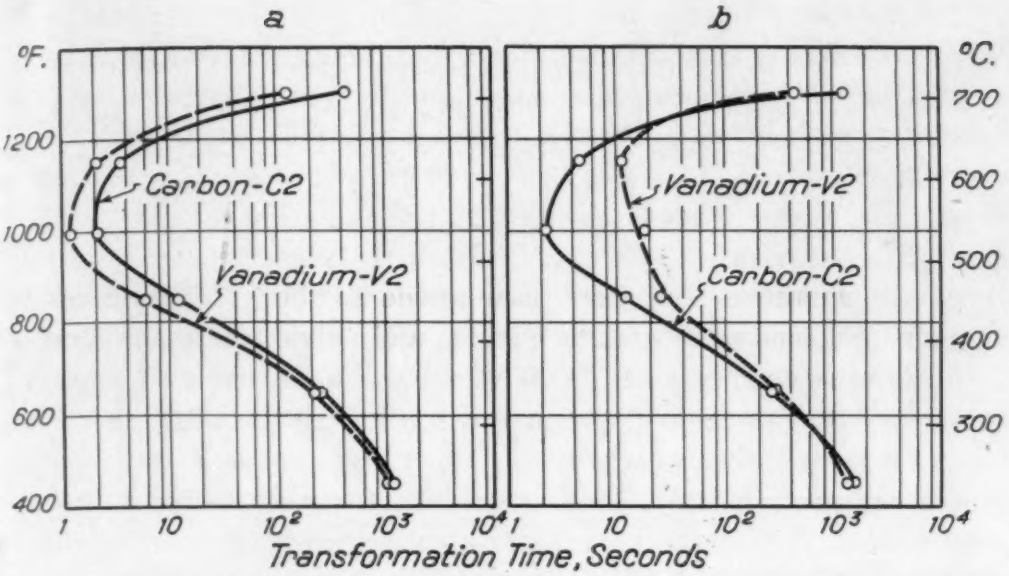


Fig. 5a—Comparative Time Intervals for 50 Per Cent Transformation in Carbon and Vanadium Steels, C2 and V2 Respectively, as Quenched from 1500 Degrees Fahr., Resulting in G. S. 5-6 and G. S. 11 Respectively.

Fig. 5b—Comparative Time Intervals for 50 Per Cent Transformation in Carbon and Vanadium Steels, C2 and V2 Respectively, as Quenched from 1625 Degrees Fahr., and 1850 Degrees Fahr. Respectively, Austenitic G. S. 4-5 in Each.

That this method gives definite indications of the fundamental influence of vanadium is illustrated by reference to the following charts. Consider, for example, Fig. 4b which represents the rate of transformation of the vanadium steel V2 as affected by progressively higher heating temperature. First of all, as in the instance of the carbon steel (Fig. 4a), the increasing grain size displaces the transformation curve progressively toward the right (increased time interval) as has been pointed out previously. In addition to this the retarding effect of the coarse grains developed by the higher heating temperature is further augmented by the solution of the vanadium-rich carbides in the austenite, thereby increasing the effective alloy content of that phase and consequently making it more sluggish in initiating transformation. Associated with this increased alloy content there is a rise in the "nose" or temperature region of most rapid transformation (A′ zone), the net effect of which, in this instance, is to shift the C-shaped portion of the curve upward so that the region of most rapid reaction is now around 1150 degrees Fahr. (620 degrees Cent.) instead of about 1000 degrees Fahr. (540 degrees Cent.). This elevation is in part indicative of a higher Ae_1 for the vanadium steel when the heating temperature is such as to permit the vanadium to go into solution in the austenite.

A further confirmation of the effect of vanadium in raising the Ae_1 temperature was obtained by determining the transformation rate of the carbon and of the vanadium steels at a temperature calculated to be just below the Ae_1 for a eutectoid carbon steel. Heating temperatures were so selected as to permit at least partial solution of the vanadium-rich carbides, and at the same time to develop identical grain size in both steels. With the same procedure as before, determinations were now made at the 1325 degrees Fahr. (715 degrees Cent.) temperature level, and while the vanadium steel transformed completely in about 9000 seconds, the carbon steel reaction progressed at a slower and slower rate until at about 300,000 seconds when the transformation was about 95 per cent complete the rate was practically zero. It was obvious, therefore, that this temperature was very close to the equilibrium transformation temperature (Ae_1) for the plain carbon steel (since a reaction proceeds more slowly as the equilibrium temperature is closely approached) but somewhat below that for the vanadium steel.

In view of the higher Ae_1 it might be anticipated that regardless of the influence of coarse grains and increased austenite alloy content, both of which tend to retard the rate at other subcritical levels, the transformation rate of the vanadium steel would be greater than that of the carbon steel at temperature levels near the Ae_1, whenever the prior heating temperature is high enough to effect at least partial solution of the vanadium. That this is indeed the case may perhaps be most easily demonstrated by observing the changes in the curves in Figs. 5 and 6. Fig. 5a compares the transformation rate of steels C2 and V2 as quenched to various subcritical temperatures from 1500 degrees Fahr. (815 degrees Cent.), at which practically all the vanadium carbide remains undissolved, thus both lowering the effective (dissolved) carbon content and excluding the vanadium from solution. Consequently since grain growth is restricted in presence of these carbides, the grain size of the vanadium steel is much finer than that of the carbon steel, and accordingly under these circumstances the transformation of the vanadium steel is faster at all temperature levels. Furthermore it will be noted that the nose of each curve now lies on the same level, approximately 1000 degrees Fahr. (540 degrees Cent.).

Proceeding now to Fig. 5b in which the respective heating temperatures are such as to develop the same grain size in both steels, it will be seen that the nose of the vanadium curve has been displaced

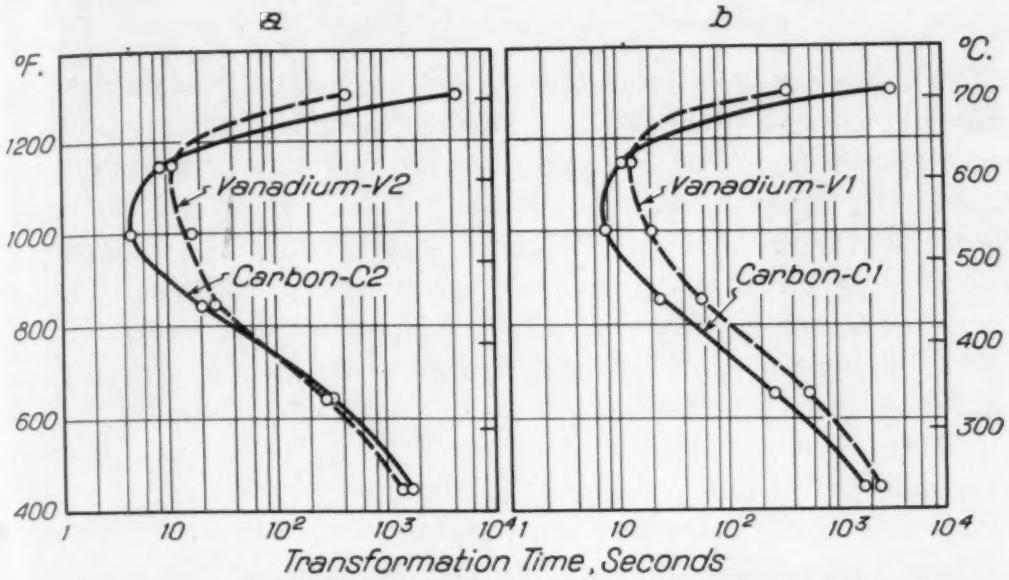


Fig. 6a—Comparative Time Intervals for 50 Per Cent Transformation in Carbon and Vanadium Steels, C2 and V2 Respectively, as Quenched from 1850 Degrees Fahr., Resulting in G. S. 2-3 and 4-5 Respectively.

Fig. 6b—Comparative Time Intervals for 50 Per Cent Transformation in Carbon and Vanadium Steels, C1 and V1 Respectively, as Quenched from 2000 Degrees Fahr., Resulting in Austenitic G. S. 2-3 in Each.

to the right and upward relative to the carbon curve; and that the curves actually cross at a higher level, above which the vanadium steel transforms more rapidly than the carbon steel of identical grain size. When both steels are quenched from the same temperature, 1850 degrees Fahr. (1010 degrees Cent.) as in Fig. 6a, under which condition the carbon steel is coarser grained than the vanadium steel, there is a greater difference in rate at the higher levels. When both steels are heated at a temperature so high that both have coarsened to the same grain size, the corresponding curves are as illustrated in Fig. 6b, where again the vanadium steel transforms faster at the higher levels, though more slowly at the lower levels. It should be observed, however, that throughout all these ranges of grain size and heating temperature, the position of the nose of the transformation curve for the plain carbon steel in contrast to the rising nose of the vanadium curve, remains fixed at approximately 1000 degrees Fahr. (540 degrees Cent.), moving only horizontally as affected by grain size.

The differences between the corresponding curves in the several figures cannot be attributed entirely to differences in the Ae_1 temperature. Indeed we believe this to be a subsidiary factor, and that the dominant factor is to be sought in differences of carbide solubility between the two types of steel.

Behavior of the Vanadium-Rich Carbides

It has become increasingly evident from this and other studies that the transformation of austenite to ferrite and carbide proceeds in a manner dependent upon the actual temperature at which it takes place. If, for example, in a eutectoid steel, the transformation takes place above the "nose" of the curve (i.e. above 1000 degrees Fahr. (540 degrees Cent.) for the plain carbon steel) ferrite and carbide separate simultaneously in a lamellar or pearlitic distribution or pattern. Below the nose, however, it would seem that this tendency to form alternate lamellae is suppressed, and instead, the change tends to sweep across a grain in intermittent plate-like or acicular zones simultaneously with, or only slightly in advance of, carbide separation; and this trend becomes more and more marked as the transformation temperature is lower. Finally the martensitic region is attained, where the carbon is probably held largely in unstable (supersaturated) solid solution and the migration or separation of carbon is prevented.

Obviously, then, in considering those temperatures at which transformation proceeds by a process of nodular growth from nuclei, it is evident that as long as a considerable portion of the carbide is present as individual particles in the undissolved condition it thereby presents just so many additional loci at which transformation may start. Comparing again the photomicrographs of Figs. 1 and 2 (which represent the steels C2 and V2 as quenched directly into brine from 1650 degrees Fahr.) (900 degrees Cent.), one is again reminded of the exceedingly large number of carbide particles in the vanadium steel in contrast to the essentially carbide-free condition of the carbon steel. It is small wonder, then, that with moderate heating temperatures where these particles are still undissolved, the vanadium steel, showered throughout with potential nuclei, should begin to transform more quickly than the corresponding carbon steel.

A typical illustration of the difference in number of nuclei furnished by the carbide particles is afforded by the photomicrographs of Fig. 7, representing a comparison of steels C2 and V2, partially transformed at 1000 degrees Fahr. (540 degrees Cent.). Different heating temperatures were used so that the grain size developed in each steel was the same, and specimens were chosen with approximately the same extent of transformation. The wide difference in the number of areas of initial transformation is easily discernible al-

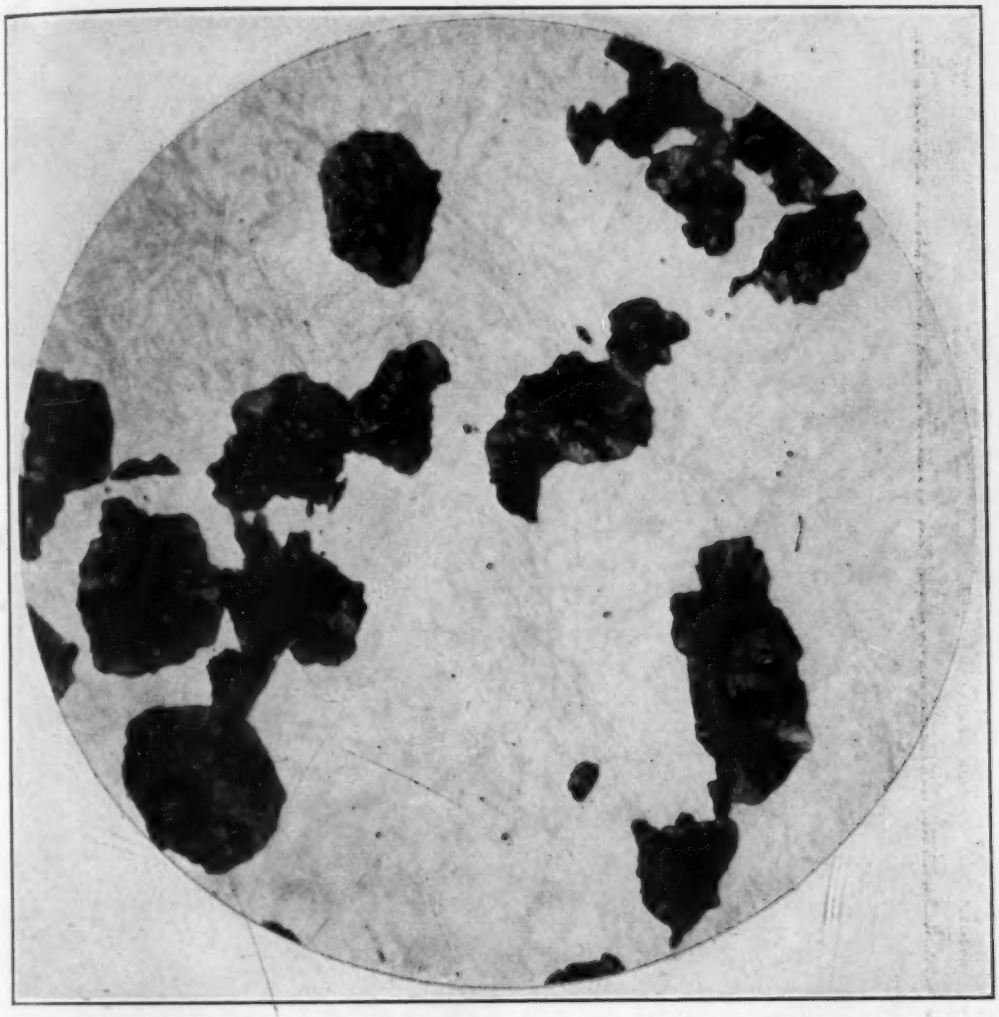


Fig. 7a and 7b—Difference in Nucleation Between Carbon Steel C2 and Vanadium Steel V2, Partially Transformed at 1000 Degrees Fahr. Austenitic Grain Size 4-5 in Each. × 1000. Fig. 7a—Carbon Steel C2. (cf. Fig. 7b.).

though at the heating temperature for the vanadium steel the carbides were already partially dissolved (thus lessening the number of possible nuclei), and this difference was much more pronounced when the vanadium steel had been heated at the same temperature as the carbon steel, for then most of the vanadium-rich carbide particles were still undissolved and therefore the number of particles available as nuclei was exceedingly large.

It was observed that the vanadium steel under certain treatments precipitated carbide particles from solid solution when the austenite was cooled to transformation levels; but these particles are not as important in their influence on transformation rates as the wholly undissolved carbide particles. It should be noted, however, that this pre-

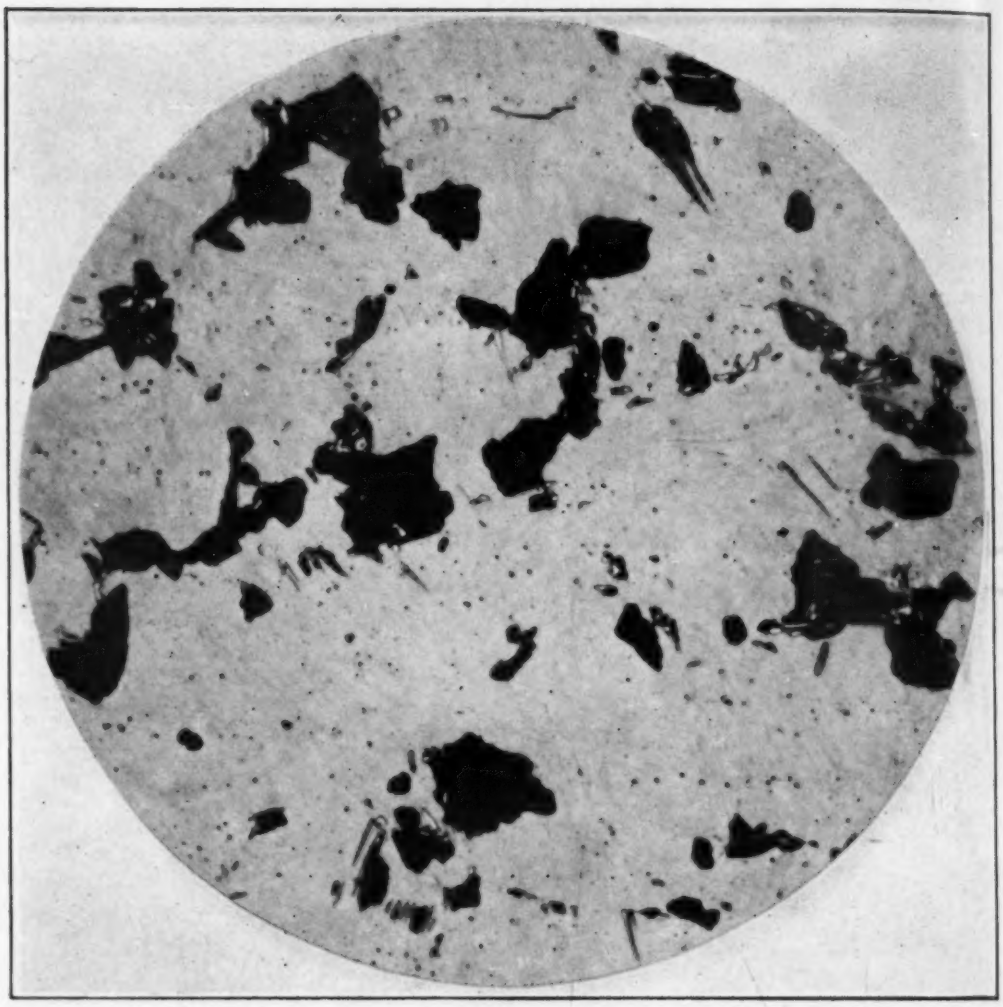


Fig. 7b—Vanadium Steel V2 Partially Transformed at 1000 Degrees Fahr. (cf. Fig. 7a.)

cipitation was apparent only in those cases in which the heating temperature was sufficiently high to dissolve a substantial proportion of the vanadium-rich carbides, the subsequent precipitation of which occurred predominantly at the higher subcritical levels. Moreover, it is of interest that this precipitation occurred *below* the Ae_1 and somewhat slowly, for specimens brought from 2000 degrees Fahr. (1095 degrees Cent.) and held for short intervals at the 1150 degrees Fahr. (620 degrees Cent.) and 1300 degrees Fahr. (705 degrees Cent.) levels showed no precipitated carbides after quenching subsequently into brine, but when held for somewhat longer periods at these levels the carbides made their appearance, preceding, however, the beginning of the eutectoid transformation proper.

As would be expected, these carbide particles precipitated selec-

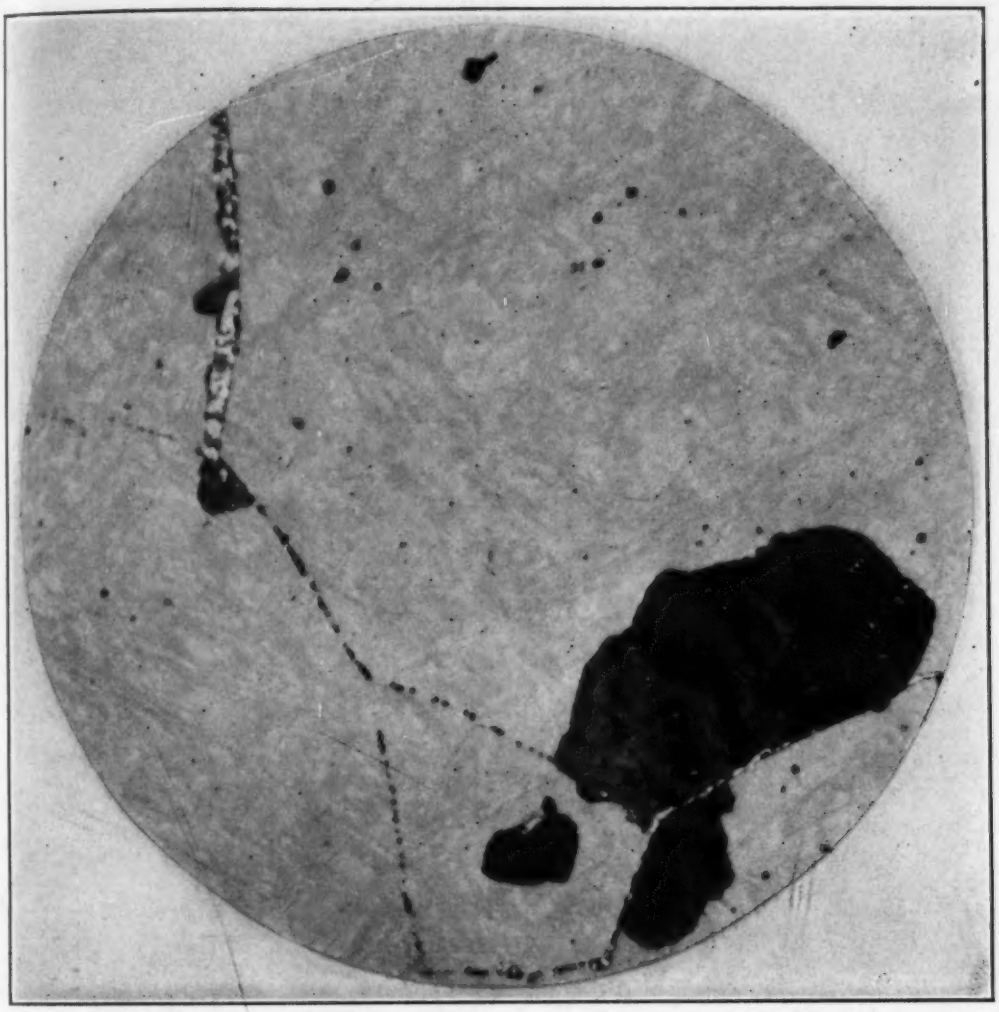


Fig. 8a and 8b—Effect of Transformation Temperature on Carbon Diffusion in Vanadium Steel V1, Partially Transformed at 1150 Degrees Fahr. and 1300 Degrees Fahr. Austenitic Grain Size 2-3 in Each. × 1000. Fig. 8a—Vanadium Steel V1 Partially Transformed at 1150 Degrees Fahr. (cf. Fig. 8b.)

tively in the grain boundaries, thereby furnishing additional nuclei in these localities, in contrast to the normal carbon steel behavior, i.e., relatively few nuclei located largely in the grain boundaries. In so doing, however, the vanadium draws locally on the carbon along the grain boundaries; this results in the development of some proeutectoid ferrite adjoining the precipitated vanadium-rich carbides in the temperature range of about 1000-1150 degrees Fahr. (540-620 degrees Cent.), in which the diffusivity of carbon is so low that carbide precipitates more rapidly than the carbon supply is replenished in the locally depleted areas. Fig. 8a, a micrograph of steel V1 quenched from 2000 degrees Fahr. (1095 degrees Cent.) and caused to trans-

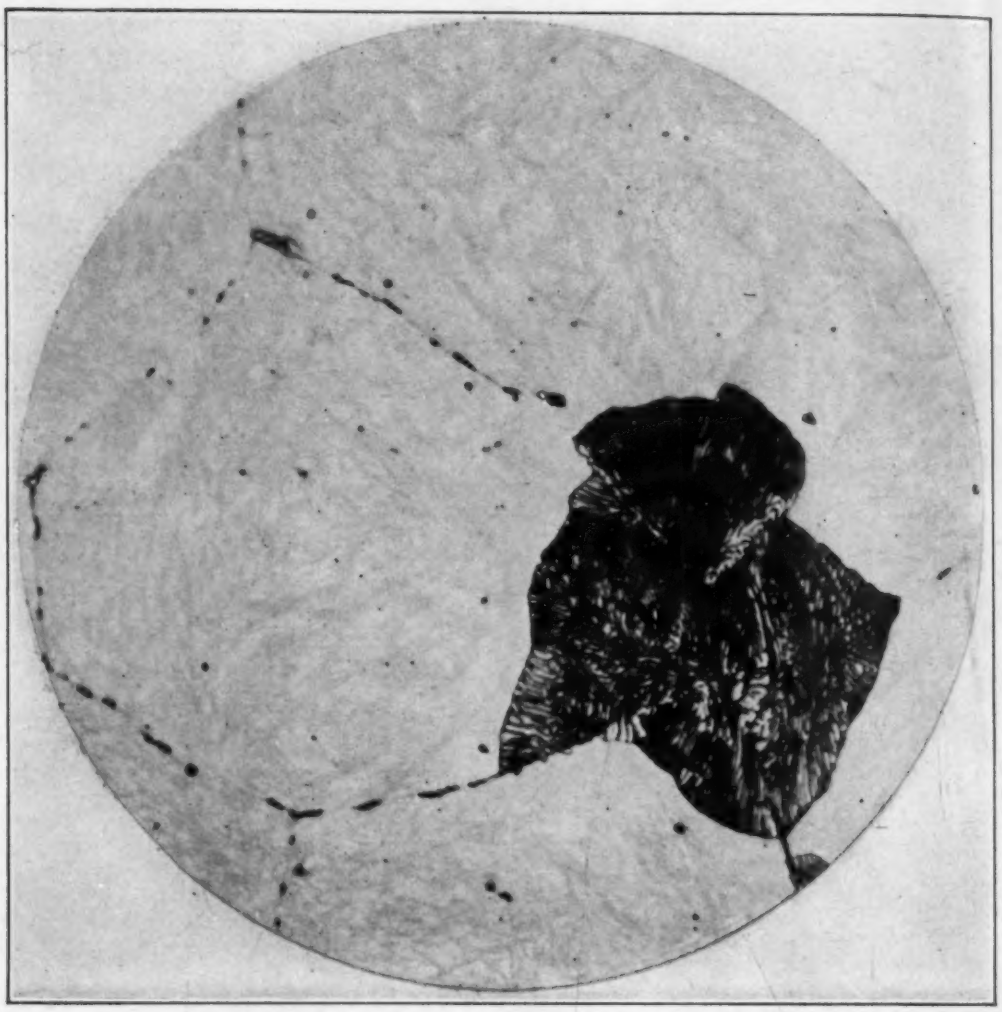


Fig. 8b—Vanadium Steel V1 Partially Transformed at 1300 Degrees Fahr. (cf. Fig. 8a.)

form to a limited extent at 1150 degrees Fahr. (620 degrees Cent.), provides an illustration of this behavior. The local depletion in carbon owing to its comparatively low diffusivity at this temperature is such that narrow bands of proeutectoid ferrite surround the carbide particles precipitated at the grain boundaries.

Fig. 8b, on the other hand, represents the same steel, also quenched from 2000 degrees Fahr. (1095 degrees Cent.) but this time caused to transform at 1300 degrees Fahr. (705 degrees Cent.). Vanadium-rich carbides have again been precipitated on the grain boundaries, but at this temperature level carbon depletion and replenishment are presumably balanced; consequently no proeutectoid ferrite is developed, since the amount of carbon involved is a relatively small proportion of the total in the steel. This precipitation of

vanadium-rich carbide is analogous to a proeutectoid carbide behavior and no doubt occurs owing to the much smaller solubility of this carbide in austenite.

Tempering

In concluding the studies of the effect of these small vanadium additions, a few tests were made to determine their influence on tempering. For this purpose disks of steels C2 and V2 were quenched directly into brine from 1750 and 1925 degrees Fahr. (955 and 1050 degrees Cent.) respectively, the size and shape of the specimens being such that a short heating period insured complete carbide solution and full martensitic hardness in each.

Specimens of each steel were subsequently tempered together (for one hour) at the several tempering temperatures, and the hardness values resulting from each treatment are plotted in Fig. 9. From this it will be observed that for tempering temperatures up to about 400 degrees Cent. (750 degrees Fahr.) the vanadium steel softens at essentially the same rate as the plain carbon steel, but that above this temperature the softening of the vanadium steel is definitely retarded. Although the difference in hardness between the two steels is not very marked for any similar tempering treatment (maximum about 10 points Rockwell C for the tempering at 600 degrees Cent. (1110 degrees Fahr.) the trend is nevertheless unmistakable.

A possible explanation of this retardation is perhaps afforded by a consideration of the conditions obtaining in the vanadium steel during the tempering process. There is, first of all, a preponderance of iron atoms (relative to the vanadium atoms) available for each carbon atom, as a consequence of which it would be expected that the first action on tempering would be a precipitation of iron-rich carbide (Fe_3C) rather than vanadium-rich carbide. Availability probably controls the initial combination, for each carbon atom is literally surrounded by iron atoms. Indeed this must be the case, for, as has been indicated in other publications mentioned previously (4), vanadium-rich carbide does not form until higher tempering temperatures are attained. These iron-rich carbides in the vanadium steel coalesce with increasing temperature, as in plain carbon steel, and the steel softens correspondingly. In fact, as can be readily seen, the softening curve of the vanadium steel very closely approximates that of the carbon steel at the lower temperatures, and this condition prevails until a tempering temperature is reached at which the mo-

bility of the carbon atoms is so increased that many of them can now migrate to vanadium atoms within the one-hour interval. At this temperature, (which depends upon the composition of the steel), vanadium-rich carbide slowly precipitates; a plausible explanation of the nature of this phenomenon suggests itself on the basis that this is analogous to the process of stabilization of the high alloy stainless steels through the precipitation of titanium carbide.

At the temperature at which the mobility of the carbon atoms is such that they become available to the vanadium atoms, the ferrite

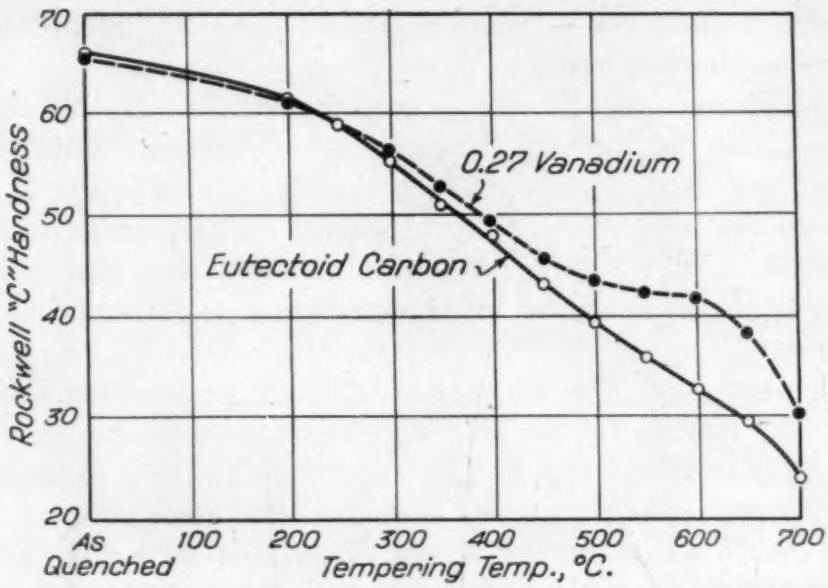


Fig. 9—Influence of Vanadium on the Hardness of Quenched Eutectoid Carbon Steel Tempered at Various Temperatures for 1 Hour.

phase is capable of holding in saturated solid solution a certain amount, however small, of carbon. This slight solubility is quite sufficient to permit migration and re-combination of the carbon, but because of the relative insolubility of vanadium-rich carbide some of the carbon is precipitated in this form; whereupon enough iron carbide dissolves to saturate the ferrite again. Thus through a continuous process based on the difference in solubility of the two carbides in this temperature range, the vanadium-rich carbide ultimately replaces substantially all of the iron-rich carbide, provided that sufficient vanadium is present.

In considering just how this process imparts a hardening effect, or more exactly a retardation of the rate of softening, it may be presumed that the vanadium-rich carbide, primarily by reason of its

small solubility and consequent low diffusivity, remains widely dispersed in the steel as effectively fine particles. Consequently, the formation of these vanadium-rich carbides imparts a definite precipitation hardness, which persists because coalescence of the particles is slow. At the higher tempering temperature the coalescence of even this less soluble carbide is of course accelerated, and, as indicated in Fig. 9, softening is then resumed.

Summary

Our observations of some of the effects of small vanadium additions to eutectoid steel, particularly of the rate of transformation, are interpreted as follows:

1. Vanadium, like molybdenum, tungsten, titanium, and even chromium, is preferentially carbide-forming, although definitely ferrite-soluble when present in amounts in excess of that combined with the carbon present. The carbides of vanadium are relatively insoluble, and therefore highly stable, at ordinary heat treating temperatures, and at temperatures high enough to effect solution, dissolve slowly. Their influence depends therefore upon the temperature and duration of heating prior to transformation.

2. At heating temperatures at which vanadium-rich carbide particles remain substantially undissolved, they inhibit austenitic grain growth and subsequently act as transformation nuclei; consequently the steel remains fine-grained, transforms rapidly and is therefore shallow-hardening and relatively tough at any hardness.

3. At heating temperatures high enough to dissolve an appreciable amount of these carbides, the vanadium entering into solution retards the transformation and to that extent promotes deep-hardening, though the steel remains fine-grained.

4. At heating temperatures at which the carbide particles are substantially all dissolved, the steel is coarse-grained and deep-hardening.

5. A vanadium steel softens during tempering less rapidly than a plain carbon steel, because of the precipitation of vanadium-rich carbide particles, which are widely dispersed and coalesce only slowly.

The authors gratefully acknowledge the courtesy of Mr. Jerome Strauss of the Vanadium Corporation of America, and of Mr. W. H. Wills of the Ludlum Steel Company, in furnishing steel samples.

Literature References

1. E. S. Davenport and E. C. Bain, "The Transformation of Austenite at Constant Sub-Critical Temperatures," *Transactions*, American Institute of Mining and Metallurgical Engineers, Iron and Steel Div., 1930, p. 117-154.
2. J. O. Arnold and A. A. Read, "The Chemical and Mechanical Relations of Iron, Vanadium and Carbon," *Journal*, Iron and Steel Institute, Vol. LXXXVI, 1912, p. 215-227.
3. H. H. Abram, "The Influence of Vanadium on Carbon Steel and on Steels Containing Nickel and Chromium," *Journal*, Iron and Steel Institute, Vol. CXXX, 1934, p. 351-375.
4. E. Houdremont, H. Bennek, and H. Schrader, "Hardening and Tempering of Steels Containing Carbides of Low Solubility, Especially Vanadium Steels." *Metals Technology*, Dec. 1934 (Tech. Pub. No. 585); *Transactions*, American Institute of Mining and Metallurgical Engineers, Vol. 116, 1935, p. 260-288.
5. E. C. Bain, "Factors Affecting the Inherent Hardenability of Steel," (Campbell Memorial Lecture). *Transactions*, American Society for Steel Treating, Vol. 20, 1932, p. 385-428.

DISCUSSION

Written Discussion: By W. H. Wills, metallurgist, Ludlum Steel Co., Dunkirk, N. Y.

The authors are to be commended for this careful study of the effect of small additions of vanadium to eutectoid steel. It would be interesting to know whether the various curves shown would be materially affected by variation of carbon content.

The vanadium steel investigated is a shock resisting tool steel of a type made by several mills in carbon ranges both above and below eutectoid composition. Common applications include chisels, rivet sets and other pneumatic tools, shear blades, punches, and cold header dies. There has been a difference of opinion as to whether carbon or carbon-vanadium steel is superior for some of these applications. The various points brought out in this paper should be valuable as a basis for further investigation of physical properties and for working out the heat treatments to get best results out of the vanadium steels.

The same reason that explains the fact that carbon-vanadium steel softens less rapidly than plain carbon during tempering would doubtless apply to a similar phenomenon noticed in the case of tungsten hot work steels. These usually contain vanadium as a minor alloy, but other elements being the same, the steel with the higher vanadium content appears to soften less rapidly in tempering.

Written Discussion: By Jerome Strauss, Vanadium Corp. of America, Bridgeville, Pa.

With their usual clarity, Mr. Bain and his associates have added another to their group of contributions on the hardening characteristics of steel and the manner in which their behavior is influenced by the presence of added ele-

ments and compounds. They have in this paper presented a very clear picture of manner in which vanadium functions during heat treatment and thereby indicated the logic of the means that have been known for retaining the advantages of vanadium, namely, fine grain size and uniform and highly dispersed carbides and at the same time securing high depth of hardening—namely, simultaneously increasing the vanadium content and the hardening temperature to the point of partial solution of vanadium-rich carbides, as practiced by Gill, and adding other carbide-forming elements as has been practiced for many years in such common types of tool steel as the chromium-vanadium and chromium-tungsten-vanadium steels and more recently with molybdenum-vanadium steels.

The authors rightfully stress the influence of the vanadium-rich carbides in restraining grain growth upon heating and in serving to form small and numerous grains upon cooling a steel in which all of these carbides have not been dissolved. In this they agree with Hougardy, Wever and Jellinghaus and other German investigators. They seem, however, to have dismissed rather summarily any possible contribution by compounds other than carbides without ample demonstration of the reason for this view. Before accepting the explanation of the behavior of vanadium as being complete, the probable part played by oxides, nitrides, etc., if they are present, should be much more closely considered. Likewise, the influence of much smaller amounts of vanadium, known to be highly effective in steels of varying compositions of both high and very low carbon, should be examined, especially in relation to the relative importance of different vanadium compounds, if present.

In closing, I should like to point out that whereas the authors, in discussing Table I, have mentioned the similarity of behavior of the two vanadium steels made by widely different practices, which is characteristic, no mention has been similarly made of the two carbon steels. Steel C1, prepared in my own laboratory, was free of any intentional grain-refining additions; if this were not true of steel C2, the results of this study might be slightly, although probably not seriously, affected.

Written Discussion: By O. W. McMullan, metallurgical department, Youngstown Sheet and Tube Co., Indiana Harbor Works, East Chicago, Ind.

The authors in their explanation of how vanadium inhibits grain growth apparently consider it unnecessary to seek further after noting the influence of undissolved carbides, for a satisfactory solution according to the observation made. It is well known that other explanations have been advanced and one of the authors[1] has suggested previously that the theory proposed by Jeffries regarding the mechanical obstructions of small particles in molten metal acting as crystallization centers might apply in steel where alumina particles would produce a fine grain size. The mechanism in either case would be the same except for the composition of the material forming the particles and that carbide particles would be precipitated from a solid solution rather than present in the molten steel. In either case it would seem necessary that the finely distributed material acting as nuclei for crystal formation be stable and remain

[1]E. C. Bain, "Factors Affecting the Inherent Hardenability of Steel," TRANSACTIONS, American Society for Steel Treating, 1932, Vol. 20, p. 385.

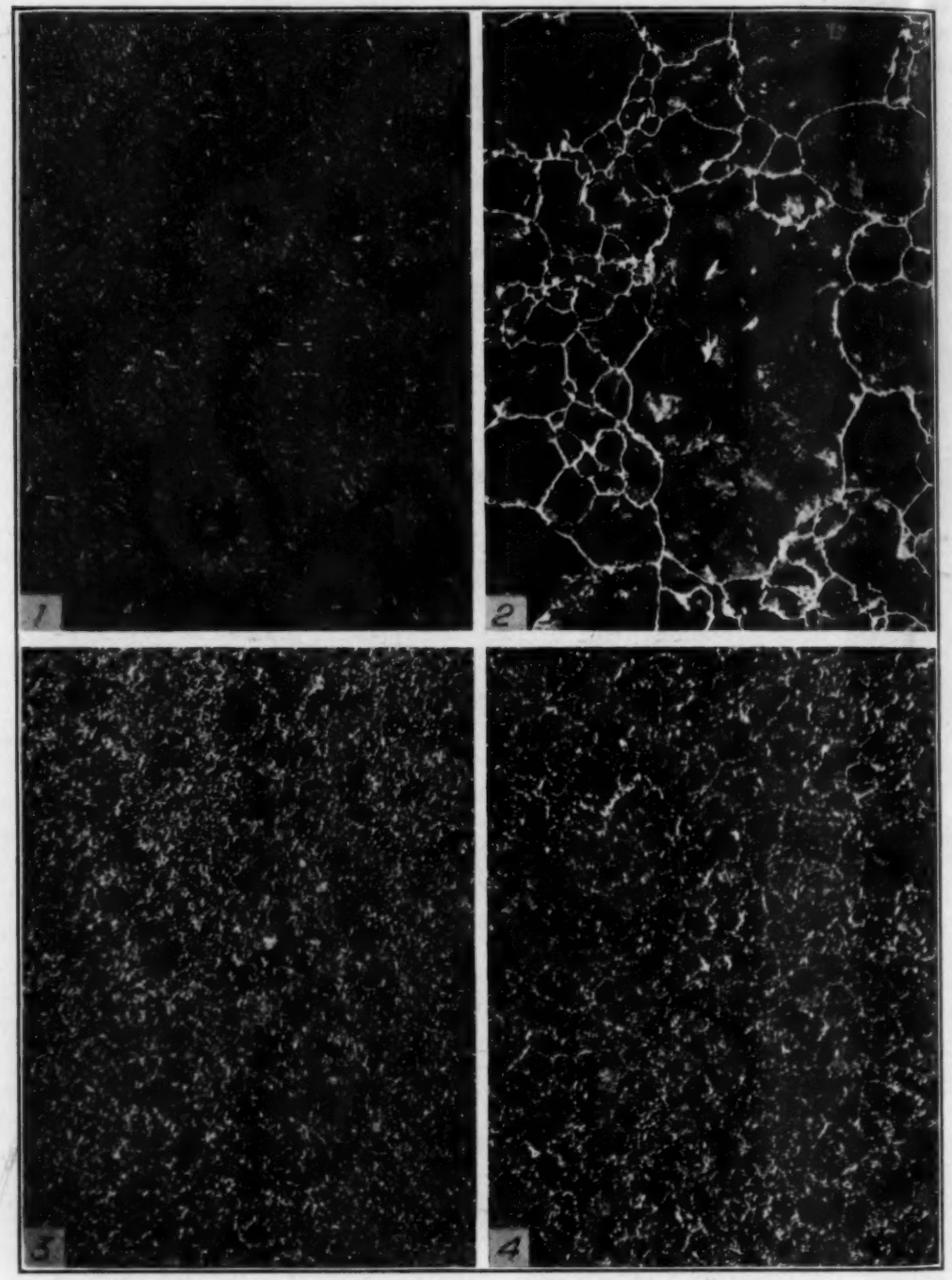


Fig. 1—Ni-Cr Steel with 1.15 Per Cent Carbon Heated 2 Hours at 2000 Degrees Fahr. and Oil-quenched. Drawn at 800 Degrees Fahr. Light Streaks are Sulphides. No Carbides are Visible. × 100.

Fig. 2—Same Steel Cooled in the Furnace from 2000 Degrees Fahr. × 100.

Fig. 3—Structure Obtained after Reheating that of Fig. 1 to 1600 Degrees Fahr. for 1 Hour and Cooling in the Furnace. × 100.

Fig. 4—Structure Resulting from Reheating that of Fig. 2 to 1600 Degrees Fahr. for 1 Hour and Furnace Cooling. × 100.

finely dispersed at the temperature of recrystallization and should it become coalesced or distributed in any massive form it would cease to inhibit grain

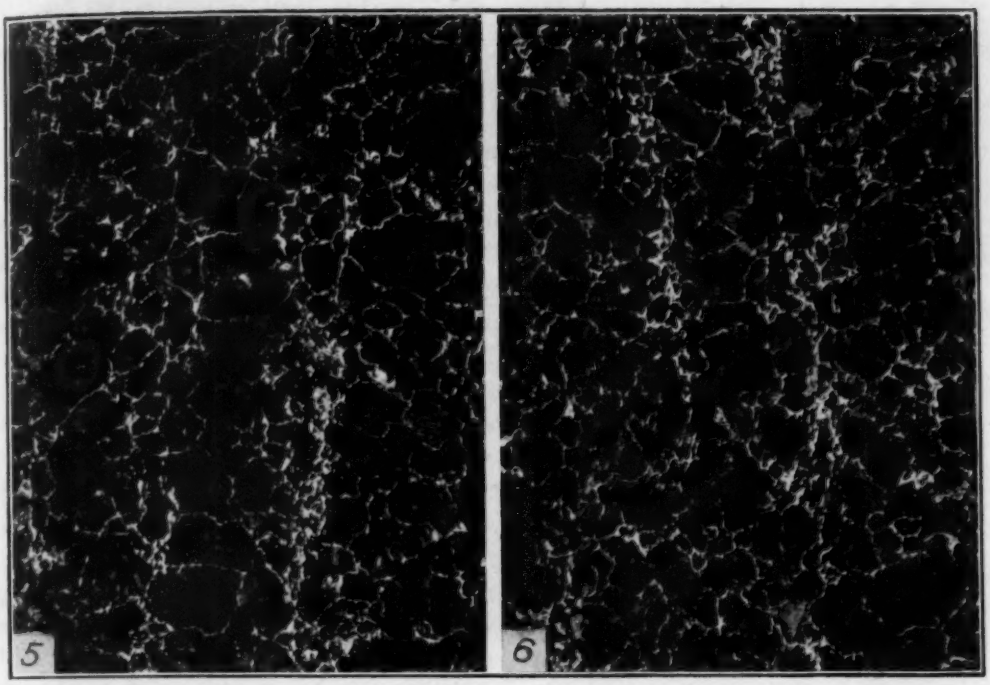


Fig. 5—Structure After That of Fig. 1 Was Reheated to 1700 Degrees Fahr. for 2 Hours and Furnace-cooled. × 100.

Fig. 6—Structure After That of Fig. 2 Was Reheated to 1700 Degrees Fahr. for 2 Hours and Furnace-cooled. × 100.

growth. The writer proposes to show that all carbides at least are not such materials, and grain size cannot be satisfactorily explained by their action.

Vanadium and aluminum are two elements which behave similarly in steel as far as the influence of small amounts on grain size is concerned. The more usual explanation is that both form oxides which are critically dispersed and act as grain centers. Vanadium also forms carbides which the authors have shown to be difficultly soluble. Aluminum will form carbides but little or no evidence has been published concerning their presence or absence in steel. The experiments on which this discussion is based concerned steels which were made fine-grained by the addition of 0.06 per cent aluminum but it is believed that the conclusions arrived at would apply equally well in the case of vanadium steels. Two melts of low nickel-chromium steel with 0.85 per cent manganese were tested. One melt contained 1.15 per cent carbon and the other 0.91 per cent. Results were similar on both heats but the carbides were more readily observable in the higher carbon heat and it is the one shown in the photomicrographs accompanying this discussion. The steel was cast into a small ingot, forged and normalized. Two small bars were heated to 2000 degrees Fahr. for 2 hours, one quenched into oil from that temperature and the other cooled slowly in the furnace. The quenched bar was drawn at 800 degrees Fahr. to permit sawing. Fig. 1 shows the quenched and drawn structure. All the carbide had gone into solution at 2000 degrees Fahr. and after the quench and draw it remains so finely dispersed as not to be visible microscopically. Fig. 2 is the slowly cooled structure and shows that a coarse carbide network has been developed by carbide precipitation around the large

austenite grains. Fig. 3 is the structure resulting after that of Fig. 1 was heated one hour at 1600 degrees Fahr. and slowly cooled and Fig. 4 that from Fig. 2 after the same treatment. This temperature was not high enough to dissolve all the carbides as shown by their presence in a specimen heated one hour at 1600 degrees Fahr. and oil-quenched.

It will be noticed that regardless of the size or distribution of the carbides to start with, the structures have similar grain sizes and carbide distribution and probably would have been completely similar had the temperature been above the A_{cm} point. Heating for 1600 degrees Fahr. for 4 hours had practically no further effect on the structures. Additional samples of the original quenched and drawn and slowly cooled bars were heated to 1700 degrees Fahr. for 2, 6 and 12 hours and slowly cooled. The structures after two hours are shown in Figs. 5 and 6, representing retreatments after the original quench and slow cool from 2000 degrees Fahr. respectively. No difference resulted in heating as long as 12 hours. Note that Figs. 5 and 6 are very similar even to the streaks of finely divided carbides. Regardless of the starting size and distribution of the carbides these inherent variations in different areas are clearly marked. The time employed for heating these samples was far shorter than used for the ordinary McQuaid-Ehn test; also the temperature was 100 degrees Fahr. lower when the structures of Figs. 3 and 4 were produced.

After noting the above behavior of carbides it does not seem possible that such mobile constituents can have more than a minor influence on grain size and that only at comparatively low temperatures or when the steel is heated for a short time. The extent of this influence would quite probably vary with the composition and stability of the carbides. If the carbides are already present as coarse, heavy envelopes before reheating how can they inhibit grain growth? Why should such a structure be so readily broken up and re-distributed as fine particles or network? If carbides are the controlling factor in grain growth would it not be logical to assume that the eutectoid structure within the coarse envelopes would grow into a single grain again before all the heavy boundary carbide dissolved if the temperature were high enough and the time long enough? Actually the carbides, no matter what their size or distribution, rather than being the controlling factors in grain growth are themselves compelled by some other controlling influence to seek a grain size characteristic of the material and temperature to which it is heated.

McQuaid[2] has pointed out some weaknesses of the theory of mechanical obstruction of grain growth by alumina particles by stating that the time of adding the aluminum makes a difference and that more aluminum is required to keep the grain fine when the melt is more highly oxidized. Granting these facts it is still possible that alumina particles may be the controlling factor in producing a fine-grained steel in spite of the fact that adding a larger amount of aluminum will again cause the grain to become coarse. Chipman[3] has shown a decrease in residual dissolved FeO in molten steel with decreasing temperature when aluminum is present. This must mean that more alumina

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[2]H. W. McQuaid, "The Importance of Aluminum Additions in Modern Commercial Steels," TRANSACTIONS, American Society for Metals, 1935, Vol. 23, p. 797.

[3]John Chipman, "Application of Thermodynamics to the Deoxidation of Liquid Steel," TRANSACTIONS, American Society for Metals, 1934, Vol. 22, p. 385.

is formed as the temperature drops. Such a procedure probably continues down to the freezing point or possibly in the solid state. It is quite possible that the alumina particles formed in the liquid steel are too large and too few in number to act as crystallization centers and that it may be only the Al_2O_3 formed on cooling down that is finely dispersed enough to affect the grain size. This might account for the necessity of timing the addition if insufficient aluminum were added to a highly oxidized melt and explain the coarsening effect observed in the last ingot poured because of slower cooling in the ladle and more chance for growth of larger alumina particles. Chipman's curves become rather flat, however, even with aluminum percentages as low as those usually added to make steel fine-grained which puts some difficulty in the way of accounting for steels again becoming coarse-grained with higher aluminum contents if the fine grain size is to be ascribed to the formation of fine alumina on cooling. The coarsening effect does take place with fairly low percentages of aluminum, however, and it may be possible that the metallic aluminum remaining is below the point where the sharp bend occurs in Chipman's curves. If the grain size is controlled by alumina formed during cooling, might it not be possible that when excess metallic aluminum is present it would dissolve the minute quantities of highly dispersed alumina formed and thus remove the crystallization centers and account for a coarse-grained steel being obtained with higher aluminum? The writer has observed an apparent decrease in the larger visible alumina particles in a few coarse-grained steels of higher aluminum content, but no extensive observations have been made. No published data were found as to the extent of the solubility of Al_2O_3 in aluminum.

Again referring specifically to vanadium carbides, the authors have observed an increase in grain size when the carbides become dissolved; an indication that these carbides were inhibiting grain growth. It is well known that grain growth inhibitors cease to remain such when the temperature becomes high enough. If the carbides in vanadium steels behave as in the aluminum-containing steels mentioned, and their size and distribution controlled perhaps by oxide nuclei, the nuclei may or may not function after the carbide dissolves away from them. It would be interesting to know if grain growth starts at the identical temperature necessary to dissolve all carbides or whether some restraining influence remains to be overcome by some further increase in temperature.

Written Discussion: By H. W. McQuaid, metallurgist, Republic Steel Corp., Massillon, Ohio.

This is to me a most interesting and instructive paper with the data and discussion presented in the usual clear and convincing manner of these writers.

One of the points which is stressed by the authors is that of the effect on the hardenability of the undissolved carbides which are present in the vanadium steel to a much higher temperature than is the case in the plain carbon steel. The authors have indicated that the decreased hardenability of the vanadium steel at the higher temperature is indirectly due to carbides which were not dissolved. They indicate that the entire decrease in hardenability is due to a small austenitic grain induced by the presence of undissolved carbide. It seems to me that the undissolved carbides may also play an im-

portant part in the more shallow hardening of the vanadium steel by decreasing to a considerable degree the amount of carbides which actually went into solution and thereby resulted in the formation of martensite on quenching. Judging from Fig. 1b, there is a considerable amount of undissolved carbide even at 1650 degrees Fahr. in the vanadium steel, so that when this steel is quenched we are actually quenching a steel which is essentially lower in carbon than the carbon steel without the vanadium. Just what the percentage of carbide in solution might be is, of course, difficult to determine. A series of hardenability tests on vanadium steels of different carbon content at temperatures high enough to insure complete solution of the carbide might indicate how much carbide would necessarily have to be in solution in the higher carbon steel to develop hardenability curves comparable to those shown in Fig. 2b. The fact that the austenitic grain size and the depth hardness curves do not check very well in the carbon and the vanadium steel, can, of course, be ascribed to the alloying effect of the vanadium, although the difference in the amount of carbide in solution may be a much more important effect, not necessarily connected with the austenitic grain size.

It would also be interesting to speculate on the difference between the two steels as to whether or not the difference is one of degree only. Thus it might be surmised that grain growth and deeper hardening take place in the plain carbon steel at a lower temperature primarily because the carbides are entirely in solution at a lower temperature and hence full hardness due to the obtaining of maximum martensite as a result of quenching. In other words, might we not assume that the same effect of difficult carbide solubility exists in carbon steels as well as those in which preferentially carbide-forming elements are present, but to a lesser degree, and that any change in the characteristics of the carbon steel which affect the carbide solubility also affects the hardenability?

Authors' Closure

The authors are very appreciative of the discussion which has been presented on this paper and believe that but few of the comments call for reply.

With regard to the question raised by Mr. Wills on the effect of raising or lowering the carbon content from the nominal eutectoid value in a steel containing about a quarter of one per cent vanadium, we would expect that if the vanadium is partially or wholly dissolved, a decrease in carbon content within rather narrow limits might tend to shift the nose of the transformation temperature-time curve somewhat to the right with concomitant increase in depth of hardening. This action appears likely since the steel is already hypereutectoid with respect to the vanadium-rich carbide and reasoning from the corresponding behavior in carbon steel a change in carbon content toward the ternary eutectoid should lessen the number of nuclei and thereby retard the initiation of transformation. On the contrary, one would expect that an increase in carbon content would have the reverse effect through increasing the amount of proeutectoid carbide.

If, as Mr. Strauss remarks, the authors appear to dismiss summarily any possible contribution of vanadium compounds other than carbide, it is certainly unintentional. They have consistently maintained an open mind on this ques-

tion and have been on the alert for any valid evidence of the influence of such compounds in these steels, which, incidentally, were reported to have been thoroughly deoxidized before any vanadium additions were made. Throughout the investigation all observations pointed to the action of a vanadium-rich carbide, and if vanadium oxide or nitride were present to any extent they appeared to exert no significant influence. It is well known that small amounts of vanadium when added to partly deoxidized steels function through the formation of these latter types of compounds, but the types of steels herein investigated do not appear to belong to this group.

The fact that the grain size pattern of steel C2 was identical with that of C1 between 1500 and 1800 degrees Fahr. and that the two did not greatly differ below and above this range indicates a close similarity in behavior from widely different steel-making practices. A comparable similarity in behavior was noted for the two vanadium steels consistent with the lesser solubility of the carbide phase in the steel containing the higher vanadium content.

Mr. McQuaid rightly calls attention to the influence of undissolved carbides in decreasing hardenability by reducing the carbon content of the austenite. The authors were not unmindful of this factor and devoted considerable thought to its evaluation. It appears that one may be led to some error in judging the amount of carbon tied up owing to the circumstance that the specific volume of vanadium carbide is much greater than that of iron carbide. The total amount of carbon which could remain undissolved as vanadium carbide is much less than 0.1 per cent in a steel containing about 0.25 per cent vanadium, and it seems unlikely that this small carbon impoverishment in the austenite could have accounted to any considerable extent for the observed behavior.

It would appear that the bulk of Mr. McMullan's remarks, interesting though they are, scarcely apply to this investigation since the steel he describes differs so widely in composition and deoxidation practice from those discussed in this paper. The steel used by Mr. McMullan contained no very strong carbide-forming element and the presence of alumina particles of appropriate size presumably accounts for its resistance to grain growth. In response to his inquiry whether grain growth starts at the temperature necessary to dissolve all carbides, it may be noted in Table I that grain growth parallels the degree of dissolution of the vanadium-rich carbides and that between 1800 and 1900 degrees Fahr. (the temperature interval corresponding to practically complete solution of the carbide phase), the steel changes sharply from a fine-grained to a coarse-grained texture. In many steels the authors have observed the simultaneous beginning of marked coarsening and the disappearance of the last substantial amount of carbide.

The authors do not intend to convey the idea that fine grain in itself is the fundamental cause of the shallow hardening behavior, but believe that the fine-grain condition is a reflection of an inherent property in the steel which is manifested by grain growth inhibition and an increased number of transformation nuclei on cooling.

EFFECT OF TITANIUM ON SOME CAST FERROUS AND NONFERROUS METALS

By Joseph A. Duma

Abstract

The use of titanium for degasifying and for cleansing steel, and as an all around "corrective agent" for such solidification defects as blowholes, segregation, mechanical enclosures, and pipe, is generally well known; but its use and its effects as an alloying element are not so widely known. This paper is an attempt at an evaluation of the alloying effects of titanium upon the mechanical properties of certain cast metals, namely—low and medium carbon steel, cast nickel-chromium alloy steel, copper-bearing steel, 18-8 stainless steel, and two copper-nickel alloys (straight and inverted monel metals). The paper besides discussing the influence of titanium on the mechanical properties of the above metals also notes its effect upon their microstructure, their weldability, general corrodibility, machinability, and age-hardenability. Photomicrographs are given of titanium-treated steel structures; also tables giving physical properties.

IN an effort to find an inexpensive high strength alloy possessing all the welding qualities of soft steel, of the Grade Bw type[1] for example, one of the first of a legion of promising alloying elements which suggested itself for investigation was titanium. Three well established facts concerning its behavior in welding were favorable to, and finally influenced the choice for its selection. Briefly, these are:

(a) Titanium is able to deprive certain air-martensitic chromium steels of their air hardening tendencies (2)[2].

(b) The presence of titanium in 18-8 stainless steels stabilizes them against "weld decay", i.e., against intergranular

[1]Composition of Bw (maximum): C = 0.30, Mn = 0.75, P = 0.05, S = 0.06, Si = 0.40, Cu = 0.25, Ni = 0.40. Other elements (each) = 0.25. The sum of the nickel and manganese shall not be greater than 1.00 per cent. Taken from Navy Department Specifications 49Sli.

[2]The figures appearing in parentheses refer to the bibliography appended to this paper.

A paper presented before the Eighteenth Annual Convention of the Society held in Cleveland, October 19 to 23, 1936. The author, Joseph A. Duma, is junior metallurgist, Norfolk Navy Yard, Portsmouth, Va. Manuscript received June 8, 1936.

chromium-rich carbide precipitation with its attendant embrittlement and loss of corrosion resistance at the grain boundaries (3), (4).

(c) Titanium, whether it be present in the coating formula or in the core of welding rods, by virtue of its arc stabilizing power, reduces sputtering and arc blowing (5).

Accordingly, a series of nickel-chromium steels of the S.A.E. 3130 analysis, to which varying amounts of titanium were added, was prepared for study. This led to the manufacture and investigation of some low and medium carbon steels containing from a trace to 1.30 per cent titanium. The properties of titanium-bearing stainless steels were next investigated, then finally those of titanium-treated monel metals.

Melting Practice

The steel as well as the nonferrous heats were all made in a basic-lined Moore "Lectromelt" electric arc furnace of one-half ton rated capacity. The charge for each carbon-titanium steel heat, 1200 pounds, consisted principally of old ship plates and shapes, with some heavy foundry returns. The nickel-chromium-titanium steels were all made from a single melt of 1500 pounds of predetermined amounts of steel, 18-8 cold rolled plate scrap, ferrochromium, and nickel. One-sixth of the heat, or 250 pounds of metal, was tapped and poured into test blocks six successive times at intervals of 5 to 10 minutes each which was the time consumed in making the addition of titanium. Thus, the chemical composition of all six fractional components of this single heat was held constant with respect to all elements but two, namely, titanium and silicon. The stainless steels were made from stainless steel plate scrap, and the synthetic copper-nickel alloys from virgin metals, i.e., electrolytic copper, electrolytic nickel, together with a small quantity of charcoal comprised the initial charge. When that charge had melted sufficiently small quantities of ferromanganese, ferrosilicon, and phosphor-copper were added.

A few words are perhaps in order on the type of slag or slags under which these alloys were made. Steel heats were made under the conventional two slags with no ore additions except in the case of heat No. 1 which is low in carbon. The high recovery of silicon and manganese in all steel heats shows that there was little oxidation

even in the meltdown. Stainless steels were made under brown (limestone, ore, and fluorspar) and white (aluminum-calcium oxide) slags; monel metals, under a lime slag. All titanium additions were made in the furnace, in the form of rutile, under a highly reducing patented slag (6). A unique feature of this process, one which differentiates it from the other more or less conventional practices, is that titanic oxide (TiO_2), and not ferrotitanium, supplies the titanium to the metal.

In practice, this addition of titanium is effected by introducing under the slag, immediately prior to tapping, a weighed mixture consisting of either two parts rutile and one part aluminum; or two parts calcined titanalba and one part aluminum; or one part rutile, one part titanalba, and one part aluminum. An intense exothermic reaction ensues between the titanium dioxide and the aluminum, resulting in the simultaneous reduction and oxidation of the former and latter respectively. The reduction of titanium dioxide is so complete that chemical analysis has failed to detect even a trace of it in the slag; and on the other hand, the cleanliness of the metal with respect to alumina inclusions is such that neither microscopic nor chemical examinations have been able to detect them. These negative findings were doubly verified with positive values, i. e., the titanium content of the metal and the aluminum content of the slag were determined, and in nearly every instance the amount of each equalled that theoretically expected from the original charge. Steels treated with titanalba were found upon analysis to contain just a trace of aluminum; those treated with rutile contained appreciable amounts of residual aluminum, for instance, a nickel-chromium heat containing 1.58 per cent titanium was found to contain 0.45 per cent aluminum, and an 18-8 stainless steel heat having a titanium content of 2.10 per cent contained 0.62 per cent aluminum.

The pouring temperature range was approximately from 2750 to 2850 degrees Fahr. (1565-1510 degrees Cent.). The melter estimated the temperature by spoon test before tapping but the actual temperature was taken with an optical pyrometer as the metal poured over the spout of the furnace. Steel from each heat, except the nickel-chromium-titanium one, was poured into two test blocks, each block weighing approximately 350 pounds. The main body of the block consisted of a slab 30x9x2 inches with twelve coupons 9x1½x2 inches cast on the bottom side. Test blocks were made in dried sand molds and were poured from a bottom-pour ladle. Steel coupons

were removed from the block with an acetylene torch; stainless steel coupons were melted off with the carbon arc.

The nickel-chromium-titanium heat was poured into six test blocks, each block weighing approximately 175 pounds. Test coupons had identically the same dimensions as those given in the preceding paragraph. Straight monel heats were poured at 2700 degrees Fahr. (1482 degrees Cent.); inverted monel heats at 2375 degrees Fahr. (1300 degrees Cent.). From each melt of monel metal a small test block was poured. The riser, or main body of the block, consisted of a rectangular slab 4x2x8 inches with two test coupons 1x2x8 inches attached to its bottom side. Coupons were detached from the block with a power saw, machined into test specimens, and tested in the green or "as cast" condition.

Tests and Test Specimens

(a) *Chemical Analysis*—Chemical analysis was made using standard recognized procedure for the accurate determination of all elements. The titanium determinations were made according to the colorimetric method of Cunningham (7).

(b) *Tensile Tests*—All tensile specimens were of the standard cylindrical type, with a 2-inch gage length and 0.505-inch diameter. Specimens were pulled in an Amsler hydraulic testing machine of 50,000 pound capacity.

The yield point was found with dividers and was taken as that load which elongated the specimen beyond the 2-inch setting of the dividers. In several instances it was detected both by the method of dividers and by the usual momentary arrest of the needle in its travel over the dial.

The results listed in the tables are the average values obtained from testing duplicate specimens but when there was a large discrepancy in results of these duplicate specimens a third specimen was subjected to the same treatment and the average taken from the two giving most similar values.

(c) *Hardness Tests*—The Brinell hardness readings were taken on the grip ends of broken tensile specimens which had been machined flat on opposite sides on a milling machine. In all Brinell tests a 3000-kilogram load and a 10-millimeter ball were used.

(d) *Impact Tests*—Izod impact specimens were the standard 10-millimeter square bars, notched transversely on two adjacent sides

by a "V" cutter to a depth of 2 millimeters and an angle of 45 degrees. The notch was finish-machined with a second cutter that was frequently ground and honed with a radius of 0.01 inch on the points. Impact tests were made in an Amsler pendulum impact testing machine of 240 foot-pounds capacity, equipped with an Izod head and anvil.

Some Charpy tensile impact tests were also made. These specimens were flat, of the shouldered type, measuring on the ends $\frac{3}{8}$x$\frac{1}{8}$ inch, and cut down in the center for a distance of $1\frac{5}{16}$ inches to a width of $\frac{1}{4}$ inch, with a 1-inch gage length. The proportion of critical dimensions, therefore, was 1-2-4, that is, the width of the specimen was twice the thickness and the effective length four times the width. The specimens were $6\frac{1}{4}$ inches long and had an effective cross sectional area of 0.0313 square inch. They were broken with a Charpy tensile pendulum hammer in the same machine as the Izod specimens.

(e) *Micro-Specimens*—Specimens for microscopic examination were taken from the large ends of broken tensile bars.

(f) *Corrosion Tests*—The specimens used in the salt spray corrosion tests were of the plumb bob type and finished with dry No. 00 iron-free emery cloth. Stainless steel specimens were exposed for 2000 continuous hours to the 4 per cent sea salt spray; monel specimens, 100 hours; and steel specimens, 24 hours.

The six stainless steel heats besides having been exposed to the salt spray were also subjected to three 48-hour corrosion periods in not less than 200 ml of boiling 67 per cent c.p. nitric acid per square inch of exposed surface of the specimen. Specimens were 2x$\frac{1}{2}$x$\frac{1}{4}$ inch. They were given a smooth lathe finish and then finish-ground all over. The corrosion losses are expressed in inches penetration per month and are calculated after each 48-hour period from the formula—

$$R = \frac{43.9W}{A\,S\,T}$$

where

R = Corrosion rate in inches penetration per month
W = Weight loss in grams
A = Total area in square inches
S = Density in grams per ml
T = Time in hours (48)

(g) *Magnetic Tests*—The attractive force exerted by a magnet

suspended directly over each of the six stainless steel micro-specimens was ascertained by weighing.

Influence of Titanium on Low and Medium Carbon Steels

Titanium was alloyed with carbon steels in the amounts indicated in Table I. Theoretical computation shows that to appreciably soften carbon steel, titanium should be present in ratio of four times the percentage of carbon. If added in that proportion it will presumably combine with all the carbon present in the steel and form a carbide having the formula TiC (8), which, as this investigation will show, is entirely unresponsive or passive to heat treatment. In practice, however, due to mass effect and secondary reactions, the ratio of titanium generally required is 5 to 7 times the percentage of carbon.

In order to determine the effect of titanium upon the iron solid solution it was introduced in excess of this requirement to heat No. 1. Heat No. 2 is a simple carbon steel, included here to serve as a standard of comparison so that a better and more definite evaluation of the various titanium additions might be made. Heat No. 3 is one which had been made with ferrotitanium instead of rutile. Titanium was added to it in the form of low carbon ferrotitanium, half in the furnace just before tapping, and half in the stream as the metal ran into the ladle. The recovery was approximately 60 per cent. Of approximately the same composition as heat No. 3 is heat No. 4, made with rutile. Heat No. 5 contains titanium in the ratio of four times the percentage of carbon, which is the amount theoretically required to combine with all the carbon. The 6th heat contains five times as much titanium as it does carbon; this is the ratio recommended in practice.

Physical properties were obtained on each of these heats after three different conditions of treatment, namely: (1) the "as cast"con-

Table I
Composition of Low and Medium Carbon-Titanium Steels

Heat No.	Chemical Composition, Per Cent							
	C	Mn	P	S	Si	Ni	Ti	Source of Titanium
1.	0.04	0.14	0.021	0.006	0.53	0.40	1.26	Rutile
2.	0.30	0.70	0.032	0.010	0.28	0.30	none	.....
3.	0.33	0.86	0.019	0.017	0.47	0.45	0.18	Ferrotitanium
4.	0.29	0.67	0.023	0.005	0.27	0.45	0.20	Rutile
5.	0.27	0.91	0.021	0.009	0.27	0.11	1.05	Rutile
6.	0.25	0.33	0.023	0.008	0.29	0.11	1.30	Rutile

Table II
Physical Properties of Carbon-Titanium Steels

Heat No.	Tensile Strength Lbs. Per Sq. In.	Yield Point Lbs. Per Sq. In.	Elongation %	Reduction of Area %	Izod Impact Ft.-Lbs.	Brinell Hardness Numeral	Treatment Temp.:Hold:Cool °F. Hrs.
1	45,000	35,500	4.0	4.0	2.0	131	As Cast
2	81,400	41,600	22.0	34.5	8.0	...	As Cast
3	70,000	.....	....	....	2.0	...	As Cast
4	87,000	56,000	5.0	3.5	2.0	...	As Cast
5	71,250	46,250	6.5	5.5	3.0	...	As Cast
6	54,000	29,500	31.0	42.0	10.0	...	As Cast
1	46,500	34,750	4.0	4.0	4.5	134	1650-2-F.C.
2	78,700	44,250	28.0	45.0	20.5	156	1650-2-F.C.
3	92,000	55,500	21.5	35.0	11.0	197	1650-2-F.C.
4	80,000	50,000	25.0	42.0	21.0	163	1650-2-F.C.
5	65,000	36,000	21.0	25.0	3.0	137	1650-2-F.C.
6	53,000	25,750	30.0	44.0	2.5	107	1650-2-F.C.
1	46,750	37,000	5.0	5.0	6.0	131	1650-2-A.C. 1525-2-A.C. 1100-6-A.C.
2	79,600	49,400	32.0	53.0	36.0	163	
3	91,100	62,000	25.0	40.0	21.0	197	
4	84,750	59,000	25.0	40.0	22.0	156	Same as Heat Above
5	66,250	39,000	21.0	30.5	3.5	143	
6	52,750	25,500	25.5	31.0	2.5	116	

dition, (2) the annealed condition, and (3) the condition resulting after double normalizing and drawing. It has been shown by Armstrong (9) that of all the many dry treatments which cast low alloy steels can be given the one which most consistently develops the best combination of high impact, high yield point, tensile strength and ductility is the double normalizing treatment followed by a drawing.

The results of the tests made on the above heats are incorporated in Table II. It will be observed that both the static and impact properties of heat No. 1 are far below those ordinarily associated with alpha iron. And, like alpha iron, titanium-bearing ferrite, it appears, is incapable of physical betterment by treatment. Attention is invited to the Brinell hardness of this steel, which, contrary to expectation, remained nearly constant in all three conditions of treatment, and this despite the fact that titanium in excess of 3 per cent is known to age harden iron (10) by the precipitation of the compound Fe_3Ti, on reheating of the quenched supersaturated solid solution. Although the above steel contained less titanium than the 3 per cent normally required to induce age-hardening, it was believed that it might have age hardened somewhat, especially when there was discovered in the microstructure a small quantity of what appeared to be Fe_3Ti.

Comparing the properties of heat treated heats Nos. 3 and 4 with those of the carbon steel heat No. 2, it will be observed that the addition of 0.20 per cent titanium—whether made in the form of ferrotitanium or rutile—has raised appreciably their tensile strength, yield point, and hardness while having decreased their ductility and notch toughness only slightly. The ferrotitanium heat appears to have an edge on the one made with rutile; this difference, the writer believes, is due not to any innate superiority of the former method of manufacture over the latter, but to its slightly higher carbon and manganese contents.

When the amount of titanium is increased to 4 to 5 times the carbon content, the ratio recommended for use in stainless steels, it seriously impairs the tensile and impact properties. Heats 5 and 6, though soft and ductile as shown by Brinell tests and elongation and reduction measurements respectively, are dynamically weak.

Metallographic examination provides confirmatory and illuminating information. The photomicrographs of Figs. 1 to 11 inclusive, show the manner in which titanium influences the structure, and through it, the mechanical properties of iron and steel. They show that titanium is essentially a carbide-forming element, which if present in sufficient quantity (theoretically, four times the carbon content) will combine with nearly all the carbon in a steel as it did in Fig. 10; if present in less than this amount it will still unite with carbon, not with all of it but with as much of it as the combining or stoichiometric ratio of one to the other will allow. It is apparent then that the nature and amount of micro-constituents in any titanium-bearing structure is largely a function of the titanium content.

As an example, the structural constituents of the steels investigated may be listed, as regards to titanium, somewhat as follow:

Per Cent Titanium	Metallographic Constituents
Less than 4 times the carbon content.	Titanium carbide, pearlite, and ferrite.
Equal to 4 times the carbon content.	Titanium carbide and ferrite.
More than 4 times the carbon content.	Titanium carbide, iron-titanium eutectic alloy, and titano-ferrite.

The physical and structural characteristics of both pearlite and ferrite, needless to say, are familiar to all metallographers; those of titanium carbide, however, are not—and so here, appropriately enough, a brief, descriptive paragraph will be devoted to the subject.

Titanium carbide, as can be seen from the photomicrograph of

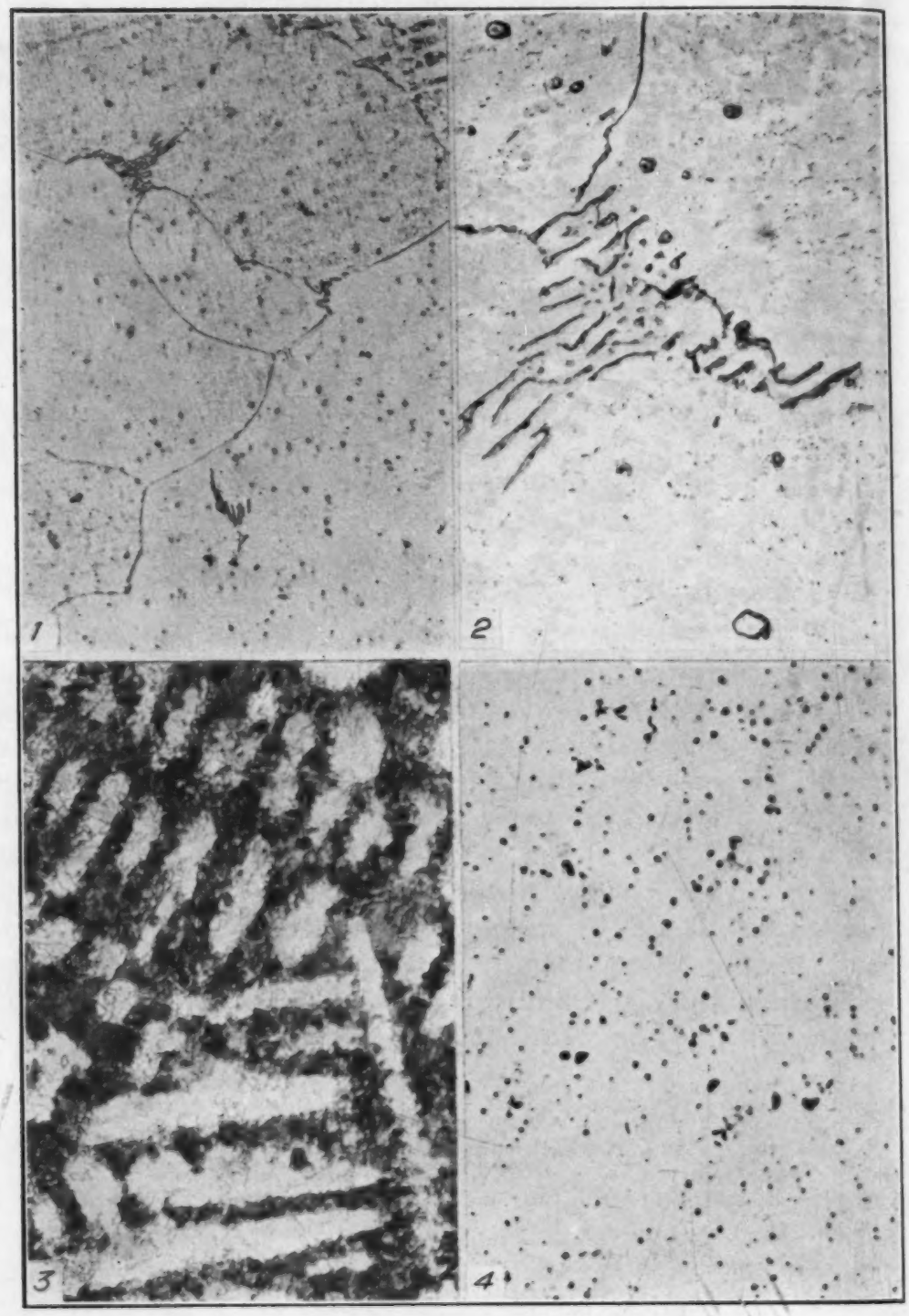

Fig. 1—Heat No. 1 Annealed. × 100.
Fig. 2—Heat No. 1 Showing Eutectic, TiC, and Ferrite, × 500.
Fig. 3—Heat No. 2 Annealed. Note the Cored Structure, × 100.
Fig. 4—Heat No. 3 Unetched. Showing Titanium Carbide Standing in Relief. × 100.

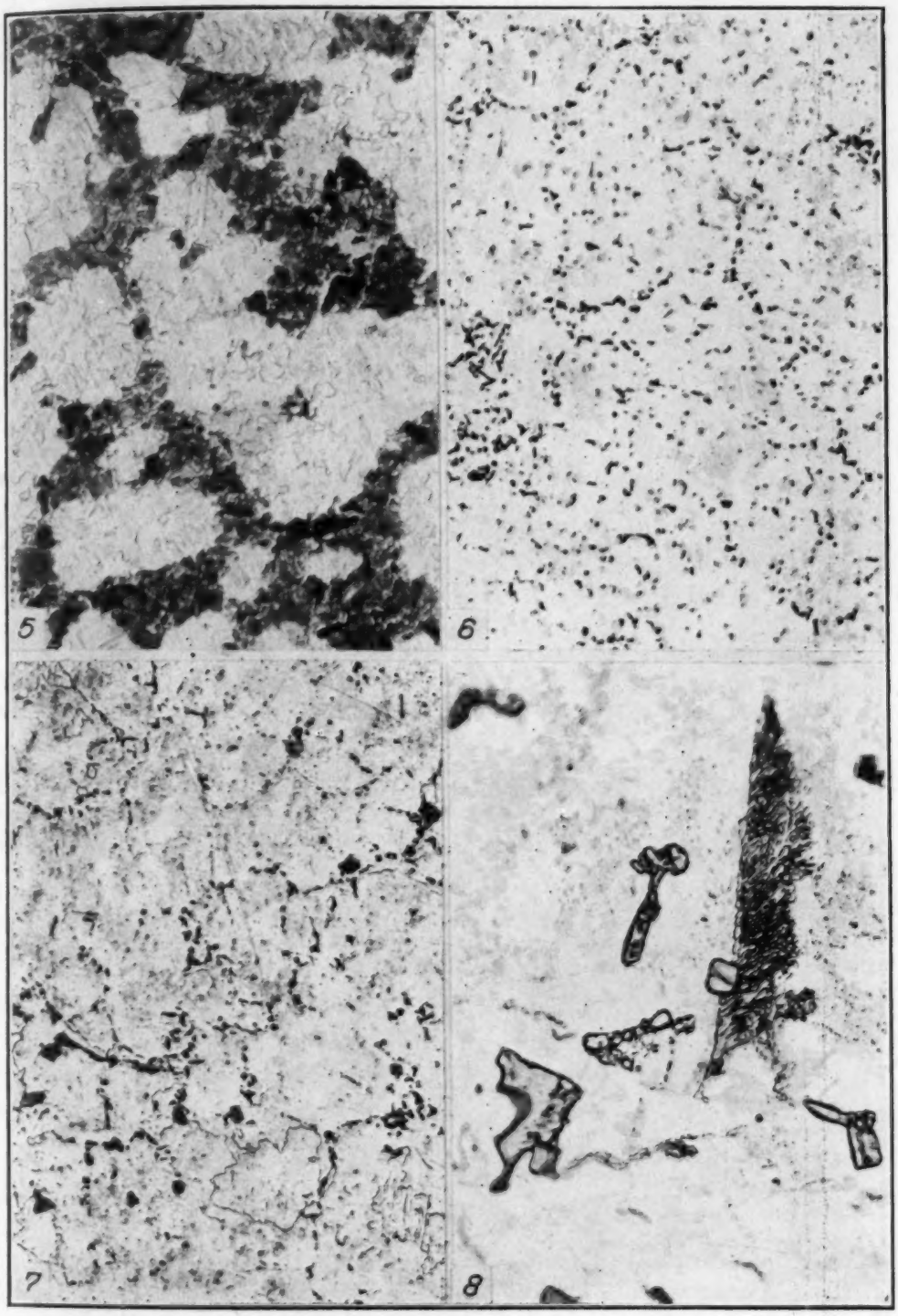

Fig. 5—Heat No. 3 Annealed. Note Absence of Coring, × 100.

Fig. 6—Heat No. 5 Unetched. Showing Titanium Carbide, × 100.

Fig. 7—Heat No. 5 Annealed. Note the Almost Complete Absence of Pearlite, × 100.

Fig. 8—Heat No. 5 Showing Some of the Small Traces of Pearlite that are Still Remaining, × 1000.

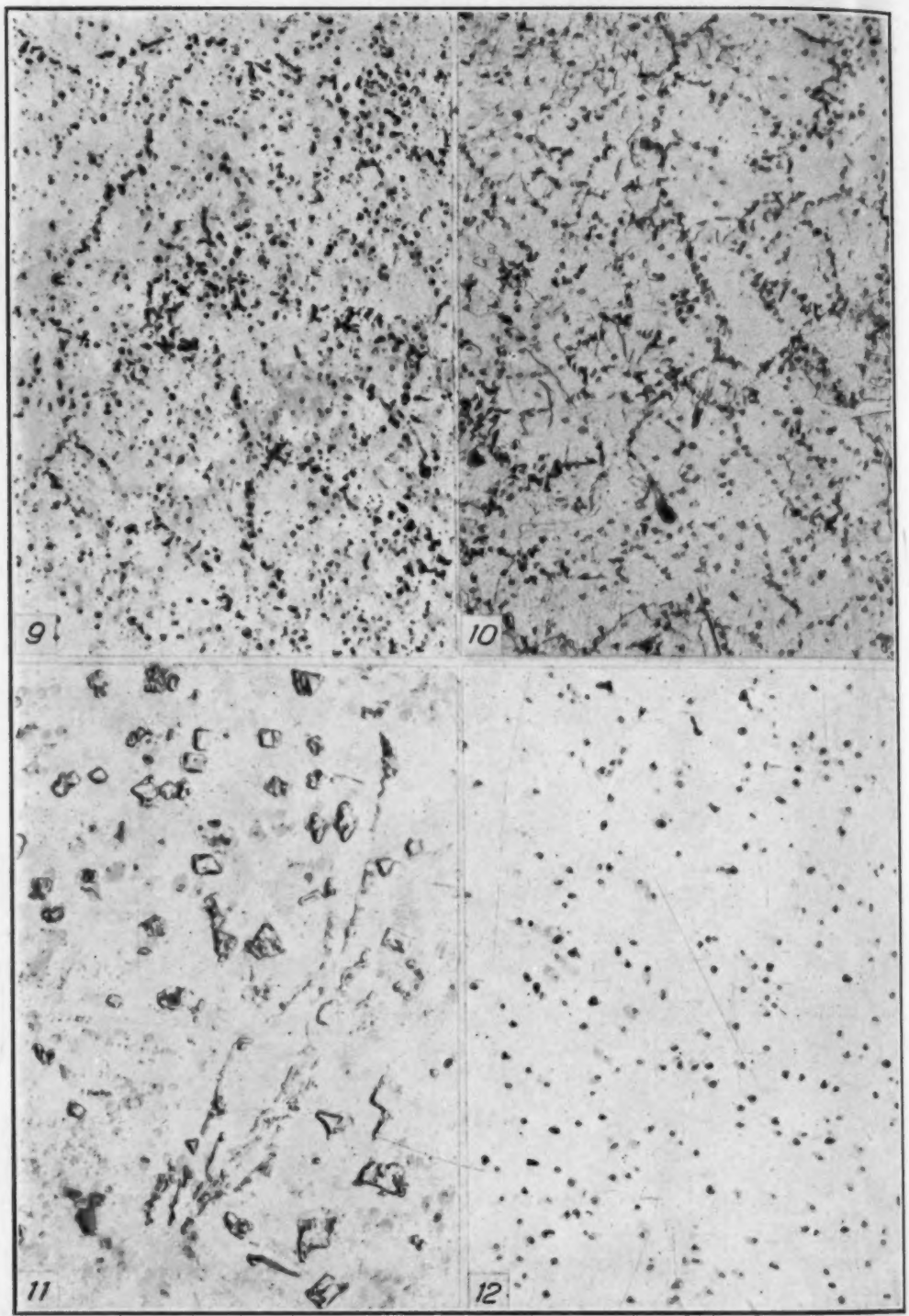

Fig. 9—Heat No. 6 Unetched. Showing Titanium Carbide, × 100.
Fig. 10—Heat No. 6 Annealed. Note Complete Absence of Pearlite, × 100.
Fig. 11—Heat No. 6 Showing Ferrite, TiC, and Some Eutectic, × 500.
Fig. 12—Heat No. 7(a)—Unetched. Titanium Carbide, × 100.

Fig. 11, is predominantly cubic in form and when viewed in white light exhibits a highly metallic luster. When present in concentrations of less than 0.50 per cent—as it is in heats Nos. 3 and 4—it manifests little if any positional preference, appearing to locate itself in more or less randomly dispersed positions; in the higher titanium steel heats Nos. 5 and 6, on the other hand, it shows a definite tendency toward segregation, embedding itself in the boundaries of primary grains. Segregations of this kind, by breaking up the continuity of the metallic matrix, cause intergranular weakness and alter adversely the impact strength of the metal. They exert these effects according to their size, distribution, and amount.

In addition to the form, and disposition, and the amount of the carbide, a few pertinent observations concerning its solubility and particle size will be made as these factors, too, are known to cause an intensive alteration in some of the physical properties of a steel. Titanium carbide, unlike any of the other metal carbides, is insoluble in solid steel even at temperatures as high as 2250 degrees Fahr. (1230 degrees Cent.). In fact, spoon tests of liquid titanium steel suddenly chilled, first in a metal mold, and then in cold water, showed undissolved carbides. Since steels are hardenable only when their carbides are soluble in solid solution, it is easy to see why those containing titanium in excess of four times their carbon content—the structural components of which are insoluble titanium carbide and ferrite, with complete absence of pearlite—are incapable of hardening. In the words of Comstock (11), "Titanium additions to low carbon alloy steel virtually serve to remove all the carbon from participation in the physical changes normally experienced in cooling, so that the net result is equivalent to a steel with almost no carbon!"

The size of the carbidic particle found in cast structures is invariably coarser than that found in wrought materials. Since treatment can neither refine, nor coalesce, nor dissolve in solid solution the carbide of titanium, modifications which ordinarily it can easily and effectively impose upon iron carbide, it cannot be used as a method of curing the embrittlement accompanying large particle size. Other methods must be resorted to for controlling or changing their size. Hot work in the form of rolling or forging is able not only to dislodge the particles from their position in the grain interfaces to other less critical situations, but also granulate them into a finer size. This explains the high ductility and impact values which have been obtained by some investigators of wrought titanium alloys.

The influence of titanium on the grain size of steel has a marked effect. The fractures of heats Nos. 6 and 1 resembled, in their brilliance and coarseness of grain, that of commercially pure zinc!

The influence of titanium additions to low and medium carbon steel may be briefly summarized as follows:

Titanium additions not in excess of 0.20 per cent are definitely beneficial to cast steel. The benefits which may be claimed for it are:

1. Comparative freedom from internal gas cavities.
2. Low nitrogen content—seldom in excess of 0.002 per cent.
3. Freedom from coring and pronounced segregation.
4. Better machinability.
5. High tensile properties with but slight diminution of ductility and notch toughness.

Titanium additions in excess of 0.20 per cent, as regards mechanical properties, have little, if any, advantageous influence. Impact resistance, tensile strength, yield point, ductility—all progressively diminish with increasing titanium content.

Nickel-Chromium-Titanium Steels

Titanium was added to nickel-chromium steel with the hope that it might make it weldable, or, what practically amounts to the same thing, non air-hardening. Nickel-chromium steel, in common with all alloy steels, when welded by the arc process will fail by cracking directly underneath the deposited metal—for the metal in this area is not only heated to above its critical range by the temperature of the arc, but is chilled by conduction and the accelerating action of the special element, or elements, so rapidly that its Ar_1 transformation point is lowered to some 400 degrees Fahr. (200 degrees Cent.), resulting in the formation of martensite and untransformed austenite. Cracks form in the martensitic zone due to the inability of martensite to resist thermal stresses generated by the unequal cooling in the deposit and parent metal. It was hoped that titanium would inhibit this formation of hard transitional constituents in the base metal. This it most certainly did do, but unfortunately to no advantage, for the high strength which was so much desired had been reduced to a value below that of a mild carbon steel.

The influence of increasing amounts of titanium upon the physical properties of nickel-chromium steel is decidedly disadvantageous

Table III
Composition of Nickel-Chromium-Titanium Steels

Heat No.	Chemical Composition, Per Cent C	Mn	P	S	Si	Ni	Cr	Ti
7	0.28	0.84	0.022	0.019	0.30	1.48	0.87	none
7(a)	0.27	0.76	0.027	0.007	0.54	1.48	0.84	0.40
7(b)	0.27	0.77	0.027	0.007	0.38	1.48	0.84	0.55
7(c)	0.28	0.75	0.027	0.007	0.33	1.48	0.84	0.60
7(d)	0.27	0.76	0.027	0.007	0.36	1.48	0.84	0.76
7(e)	0.27	0.76	0.027	0.007	0.52	1.48	0.84	1.21
7(f)	0.27	0.77	0.027	0.007	0.72	1.48	0.84	1.84

as a study of the data tabulated in Table IV will show. It will be noted that, as in the case of carbon steel, low additions increase the tensile strength, yield point, and hardness but with a more pronounced impairment of ductile and impact properties. High additions (those in excess of 0.20 per cent) are most damaging first, to impact strength, and secondly to elongation and reduction. The alloying benefits of nickel and chromium are more than nullified.

Nickel inhibits the grain coarsening tendencies of high titanium additions. All nickel-chromium-titanium steel fractures were finer grained than the carbon-titanium fractures of equivalent titanium content. The failure of all high titanium steels was distinctly intergranular in character. This is not at all surprising. Due to the presence of hard carbides forming brittle and more or less beaded membranes surrounding the grains the steel failed along the grain interfaces, the paths of minimum cohesion.

Table IV
Physical Properties of Nickel-Chromium-Titanium Steels

Heat No.	Tensile Strength Lbs. Per Sq. In.	Yield Point Lbs. Per Sq. In.	Elongation %	Reduction of Area %	Izod Impact Ft.-Lbs.	Brinell Hardness Numeral	Treatment Temp. °F.	Hold: Hrs.	Cool
7	102,750	67,500	23.0	44.0	....	217	1650	2	F.C.
7(a)	116,000	63,500	9.0	7.0	....	228	1650	2	F.C.
7(b)	94,250	58,000	12.5	14.5	....	207	1650	2	F.C.
7(c)	94,000	57,500	11.5	12.0	....	196	1650	2	F.C.
7(d)	86,000	52,250	11.5	18.0	....	187	1650	2	F.C.
7(e)	73,750	49,250	17.5	15.0	....	179	1650	2	F.C.
7(f)	70,750	46,500	22.0	22.0	....	156	1650	2	F.C.
7	103,000	69,750	18.0	35.0	28.0	222	1650	2	A.C.
							1525	2	A.C.
							1100	6	F.C.
7(a)	123,000	78,250	9.5	11.5	4.0	269			
7(b)	91,750	70,250	16.0	17.0	10.0	217			
7(c)	97,500	70,750	15.0	14.0	8.5	223	Same as		
7(d)	87,500	60,000	12.0	12.0	7.0	187	Heat 7 Above		
7(e)	71,250	52,500	6.5	5.0	3.5	170			
7(f)	57,000	46,500	3.5	3.5	2.5	153			

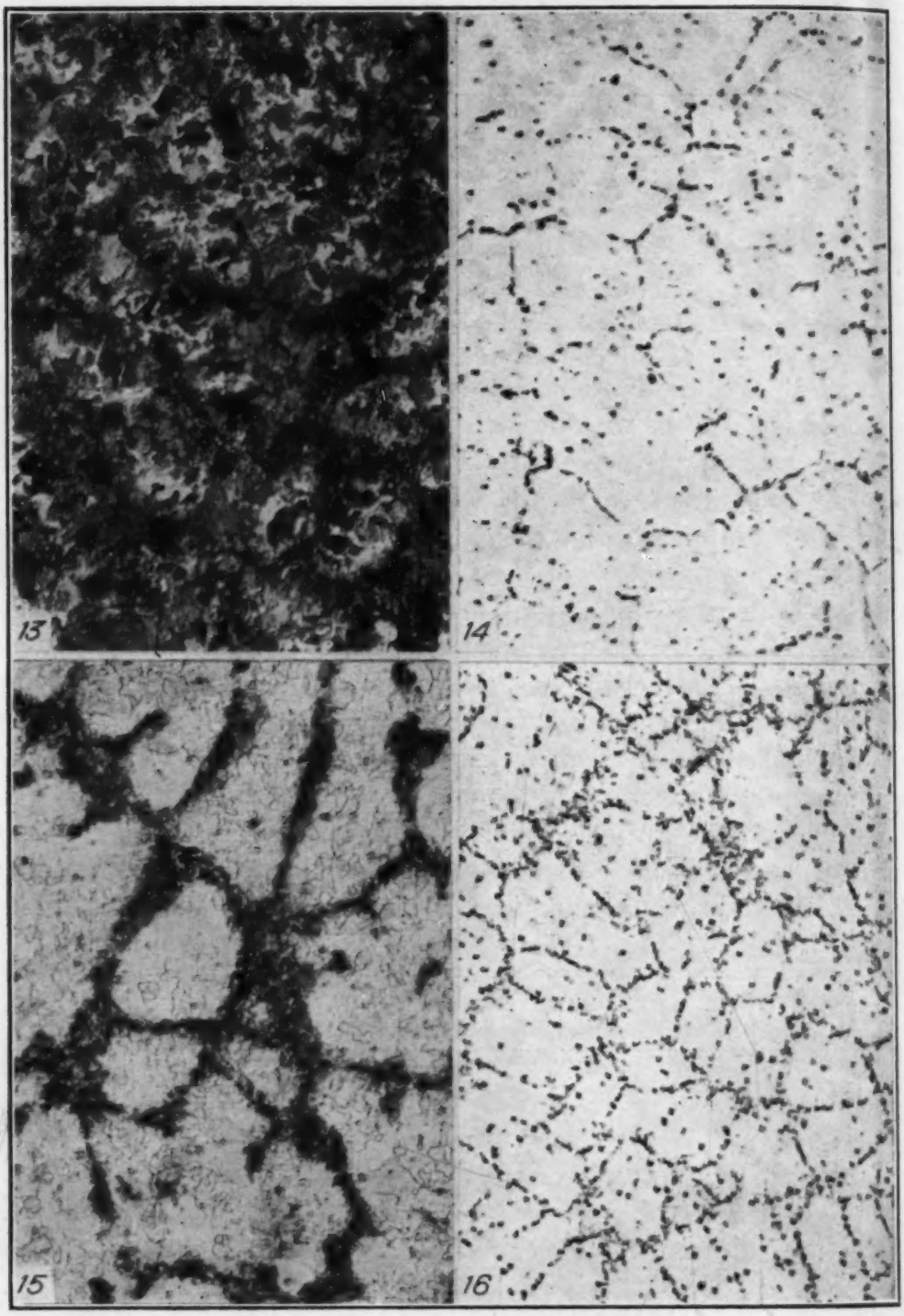

Fig. 13—Heat No. 7(a)—Annealed. Note the Amount of Ferrite and Pearlite, × 100.

Fig. 14—Heat No. 7(d)—Unetched. Titanium Carbide, × 100.

Fig. 15—Heat No. 7(d)—Annealed. Note Relative Amount of Ferrite and Pearlite, × 100.

Fig. 16—Heat No. 7(f)—Unetched. Segregated Titanium Carbide.

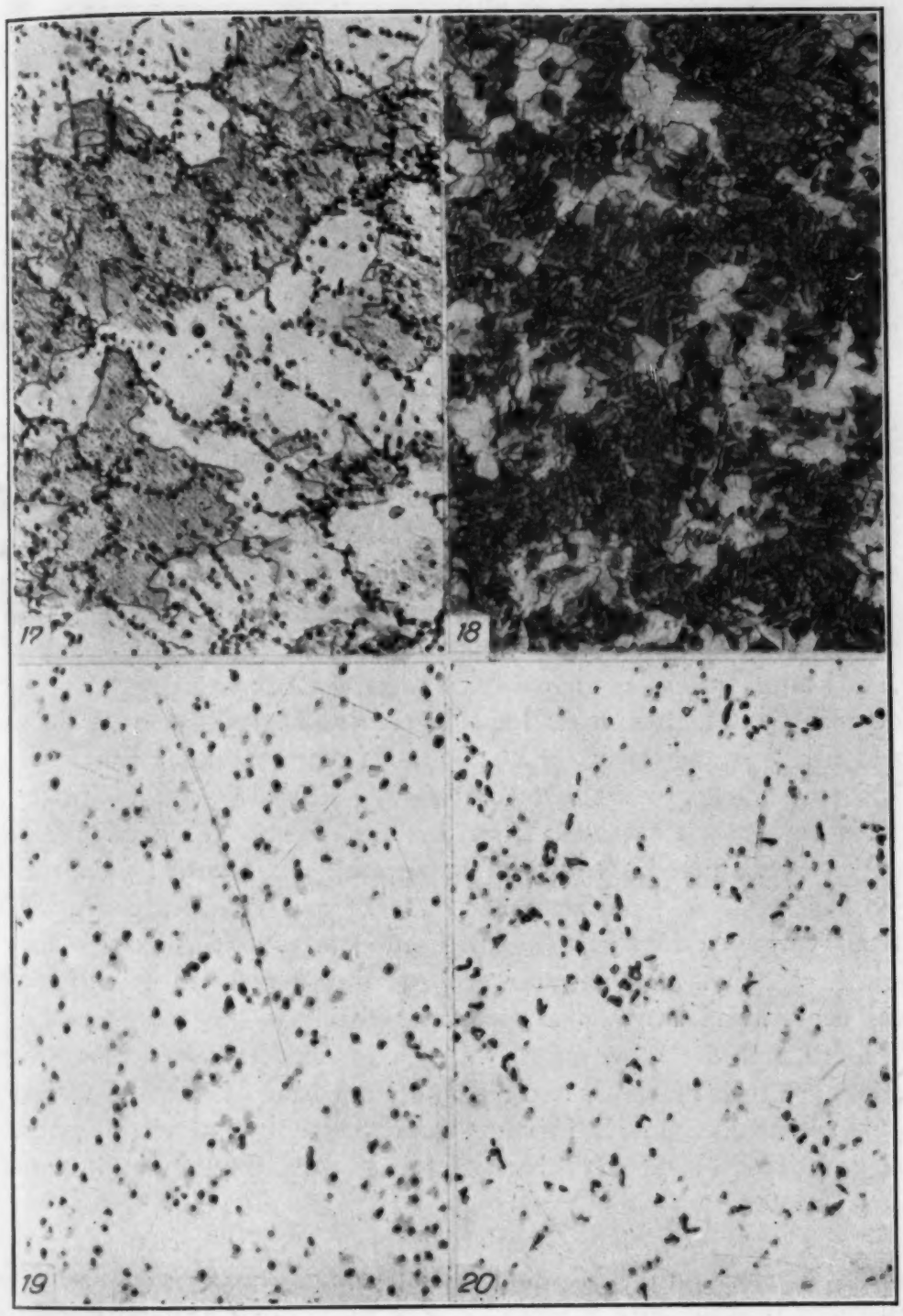

Fig. 17—Heat No. 7(f)—Annealed. Note Complete Absence of Pearlite.

Fig. 18—Heat No. 7—Annealed. Compare Amount of Pearlite in this Heat with Heats 7(a), 7(d), and 7(f).

Fig. 19—Heat No. 13—Unetched. Titanium Carbide in Quenched Stainless Steel × 100.

Fig. 20—Heat No. 15—Unetched. Titanium Carbide in Quenched Stainless Steel, × 100.

Table V
Hardness and Tensile Properties of Nickel-Chromium-Titanium Steels After Aging at Indicated Temperatures

Heat No.	Treatment					Brinell Hardness	T. S. Lbs. Per Sq. In.	Y. P. Lbs. Per Sq. In.	Elon. %	R. A. %
	Temp.	Cool	Temp.	Cool	Draw					
7(d)	1650	AC	1550	AC	700	207	95,750	62,250	9.5	10.0
7(d)	1650	AC	1550	AC	900	197	91,000	61,250	10.0	11.5
7(d)	1650	AC	1550	AC	1100	187	87,500	60,000	12.0	12.0
7(d)	1650	AC	1550	AC	1300	179	85,500	58,500	15.0	14.0
7(d)	1650	AC	1550	AC	1350	175	84,000	57,250	16.0	16.0
7(f)	1650	AC	1550	AC	700	156	59,000	47,750	4.0	3.5
7(f)	1650	AC	1550	AC	1350	156	52,000	46,250	3.0	3.5

Structural modifications traceable to titanium are recorded in the photomicrographs of Figs 12 to 18 inclusive. These views clearly demonstrate how increasing percentages of titanium alter the ratio of pearlite to ferrite, reducing the former and augmenting the latter, until at the percentage of 1.21 titanium, pearlite disappears completely from the structure.

In order to determine whether titanium in the amounts used possessed any age-hardening properties a number of bars from heats 7 (d) and 7 (f) were air-cooled, first from 1650 degrees Fahr. (898 degrees Cent.), then from 1550 degrees Fahr. (840 degrees Cent.), and finally drawn back for six hours at temperatures ranging from 700 to 1350 degrees Fahr. (370 to 735 degrees Cent.). Table V gives the results obtained from this treatment. No age-hardening effects were noted. In addition, several specimens, 1-inch round by 4 inches long, from heats 7(c) and 7(f) were quenched in water from various critical temperatures to determine their depth hardening capacity. Specimens were cut transversely in two through the center, and two Vickers hardness readings—one on the outside surface and the other on the center of the transverse cut—were taken. Table VI shows the high titanium heat 7(f) to be virtually unhardenable. This is in complete agreement with metallographic observations. Heat 7(c) hardened uniformly throughout its entire cross section.

All these steels have been subjected to the 4 per cent salt spray test. The results disclose that high titanium-bearing steels are able to resist the corrosive action of saline vapors much more effectively than either the straight carbon, straight nickel-chromium, or low titanium steels. The titanium-free steels rusted after an exposure of 2 to 4 hours; the ferrite titanium steels, on the other hand, showed no sign of corrosion until after 18 hours.

Table VI
Hardenability of Nickel-Chromium-Titanium Steels

Quenched in H_2O from Temp. °F.	Brinell Hardness — Heat No. 7(f) Surface	Heat No. 7(f) Center	Heat No. 7(c) Surface	Heat No. 7(c) Center
1400	146	146	197	197
1500	146	146	388	388
1600	153*	146	444	430
1700	153	153	444	444

*When Aged at 1350 degrees Fahr.—6 hrs. Brinell Hardness = 146
When Aged at 700 degrees Fahr.—6 hrs. Brinell Hardness = 153

Two other unusual characteristics of titanium-treated steels which are worthy of special note are: (1) their ability to take on a high polish, and (2) their ready machinability. They lend themselves easily to boring, turning, drilling, sawing, etc., always breaking off with a fine, short, crisp chip.

Summing up, titanium under 0.20 per cent affects favorably the tensile properties of nickel-chromium steel. Higher percentages lower strength, decrease ductility, and reduce its inherently high shock resisting qualities to practically nothing. A few commendable features of high titanium are: its ability to take heavier and faster rough cuts and its augmented resistance to oxidizing and corroding media.

Table VII
Physical Properties of Copper-Titanium Steel

Chemical Composition

	C	Mn	P	S	Si	Ni	Cu	Ti
Heat No. 8	0.29	0.94	0.025	0.007	0.87	0.86	1.41	0.38
Heat No. 9	0.24	1.02	0.024	0.007	0.31	0.64	1.26	none

Tensile Properties

Heat No.	Treatment — Temp.	Hrs.	Cool	T. S. Lbs. Per Sq. In.	Y. P. Lbs. Per Sq. In.	Elon. %	R. A. %	Izod Ft.-Lbs.
8	Untreated			112,000	104,000	1.0	0.8	1.5
9	Untreated			91,000	71,400	12.0	18.5	5.5
8	1650	2	F.C.	95,500	65,250	12.0	13.0	9.0
9	1650	2	F.C.	83,500	59,000	25.0	43.0	40.0
8	1650	2	A.C.	105,000	84,000	10.0	12.0	7.0
	1525	2	A.C.					
	1100	6	F.C.					
9	Same as Heat 8			87,250	72,750	25.0	57.0	29 to 47

Age-Hardening Properties

Treatment					Brinell Hardness Heat No. 8	Heat No. 9
1550	2 hrs.	A.C.	700	6 hrs.	228	187
1550	2 hrs.	A.C.	900	6 hrs.	262	223
1550	2 hrs.	A.C.	1100	6 hrs.	238	207
1550	2 hrs.	A.C.	1300	6 hrs.	205	179

Copper-Titanium Steel

Long before any of the aforementioned titanium-treated steels were made, or before any systematic study of the effects of titanium was undertaken, a copper-titanium heat of the composition shown in Table VII was cast. That is the reason for its inclusion here. For comparison, it is paired up with another heat which, except for titanium, is approximately of the same analysis.

A glance at Table VII will show that excessive titanium has the same deleterious effect on the physical properties of copper-bearing steel as it does on carbon and nickel-chromium steels. Copper-bearing steels are reputed for their high ductility and excellent impact properties (12). Copper steel heat No. 9 is no exception. Alloying them with titanium, however, embrittles them both statically and dynamically. In fact, every addition of titanium made thus far, has been accompanied by some impairment of impact properties.

The effect of titanium upon the ever disputed "red shortness" of copper-bearing steels was not investigated. Nor were there any extensive tests made on "precipitation hardening." A few hardness determinations after exposure to certain aging temperatures were the only tests taken. It will be noted that maximum age-hardening took place at 900 degrees Fahr. (480 degrees Cent.).

Copper, and not titanium, was the precipitated constituent.

Microscopic examination of the copper-titanium steel disclosed the presence of—in addition to the usual titanium carbide, pearlite and ferrite—numerous platelets of copper or high copper iron. They were also observed in the titanium-free copper steel.

18-8 Titanium-Treated Corrosion Resisting Steel

Much has been heard about titanium in connection with the prevention of intergranular corrosion in corrosion resisting steels. The disease of intergranular corrosion, which at one time was wont to destroy prematurely the serviceability of austenitic stainless steels of the 18-8 type, is now fairly well under control. True, titanium is not the only anti-toxin for intergranular susceptibility; there are others equally as effective, among them—(1) heat treatment, (2) low carbon content (less than about 0.02 per cent for complete immunity), (3) sufficient grain boundary extent, (4) cold work or

Table VIII

Composition and Mechanical Properties of Cast 18-8 Titanium-Treated Steels

Heat No.	Chemical Composition, Per Cent C	Mn	P	S	Si	Ni	Cr	Ti
10	0.12	0.62	0.017	0.008	0.41	13.15	22.00	none
11	0.11	0.59	0.015	0.006	0.51	10.39	20.29	0.11
12	0.12	0.58	0.021	0.017	0.90	10.03	22.59	0.35
13	0.10	0.74	0.019	0.006	0.79	11.40	23.91	0.46
14	0.12	0.69	0.023	0.007	0.70	11.50	22.61	0.90
15	0.13	0.68	0.023	0.007	0.68	11.54	22.49	3.50

Mechanical Properties After Quenching in Water From 2000 Degrees Fahr. (1093 Degrees Cent.)

Heat. No.	T. S. Lbs. Per Sq. In.	Y. P. Lbs. Per Sq. In.	Elon. %	R. A. %	Brinell Hardness	Cold Bend	Izod Ft.-Lbs.	Tensile Impact Ft.-Lbs.	Tensile Impact Elon. %
10	71,750	38,750	54.0	61.5	143	Flat	92.0	168	46
11	78,250	43,000	46.0	57.5	163	Flat	97.0	149	36
12	80,500	44,500	45.5	57.0	170	Flat	77.0	108	31
13	82,500	46,000	42.5	56.5	174	Flat	48.0	115	32
14	97,000	61,500	31.0	40.5	207	180°	30.0	86	22
15	137,750	100,000	3.0	3.5	302	10°	1.5	...	..

an abundance of slip planes, (5) other elements, to mention only a few, columbium, tantalum, uranium (13)—but many of these alternative remedies are not always applicatory, nor are any of them comparable to titanium (rutile) in lowness of cost. Furthermore, titanium gives permanent immunity, unimpairable by any treatment, thermal or mechanical. This is why the use of titanium as a stabilizing agent is so popular.

The influence of titanium upon the corrodibility of stainless steels has been intensively studied, so much so that its effects on their mechanical properties have been, not exactly overlooked, but not scrutinized any too critically; nor have the effects of high percentages of that element on those properties been studied.

In Table VIII are shown the composition and mechanical properties of six heats of stainless steel containing from 0.11 to 3.50 per cent titanium. Heat 10 is a comparison heat, an ordinary 18-8 material of almost identical analysis, except with respect to titanium. With the exception of heat 15, each heat given in this list represents the typical, or average properties of dozens of almost identical compositions, deliberately repeated in order to establish the degree of consistency to be expected in commercial practice. These heats are not laboratory, or experimental tests, but represent actual commercial castings produced in a basic electric furnace in melts of from 1000 to 2000 pounds each.

Reference to Table VIII shows that progressively higher concentrations of titanium result in correspondingly higher values of

tensile strength, yield point, and hardness. And again, as formerly in the case of carbon, nickel-chromium, and copper-bearing steels, this elevation of tensile strength is obtained at the expense of reduced impact and ductile properties. Due to the inherent, extreme plasticity of the austenitic alloy, additions as high as 0.50 to 0.60 per cent titanium can be made and the resultant changes will leave the material still in a class with the toughest metals. Six times the carbon content in excess of 0.02 per cent may be the correct proportion of titanium for the obtainment of maximum immunity from intergranular susceptibility but it does not necessarily follow that this also is the best proportion from the standpoint of mechanical properties. In order to secure both immunity and "balanced" physical properties the carbon should not exceed 0.10 per cent, and the titanium, 0.50 per cent.

The bend, tensile, and impact specimens from heat 15, unlike those from heats 10 to 14, broke off sharply with a coarsely crystalline fracture. To ascertain whether high titanium content or high temperature treatment was responsible for this complete lack of toughness four other treatments were tried, namely: (a) 1650 degrees Fahr. (900 degrees Cent.) water quench, (b) 1650 degrees Fahr. (900 degrees Cent.) normalize, (c) 1650 degrees Fahr. (900 degrees Cent.) anneal, and (d) 1550 degrees Fahr. (840 degrees Cent.) anneal. Scarcely any improvement in ductility, or in fracture appearance were shown; the highest elongation and reduction readings, 7 and 7.5 per cent respectively, were obtained on the specimens quenched in water from 1650 degrees Fahr. (900 degrees Cent.).

The effect of titanium on the microstructure of 18-8 stainless steel is shown in Figs. 19 to 28 inclusive. Figs. 19 and 20 are the unetched structures of heats 13 and 15 respectively. Fig. 21 is a high magnification view of the structure shown in Fig. 19. The hard, angular particles standing in relief are the familiar compound of titanium and carbon, and probably nitrogen. It will be noticed that, contrary to prevailing opinion, they do manifest a slight tendency toward localization. The structures of Figs. 19 and 20 contain nearly equal amounts of carbide, and this despite the fact that the titanium content of the latter is 7½ times that of the former. Obviously, this is because in low carbon, high titanium alloys the amount of carbide is a function of the carbon content; and conversely, in high carbon, low titanium alloys it is functionally dependent upon the percentage of titanium.

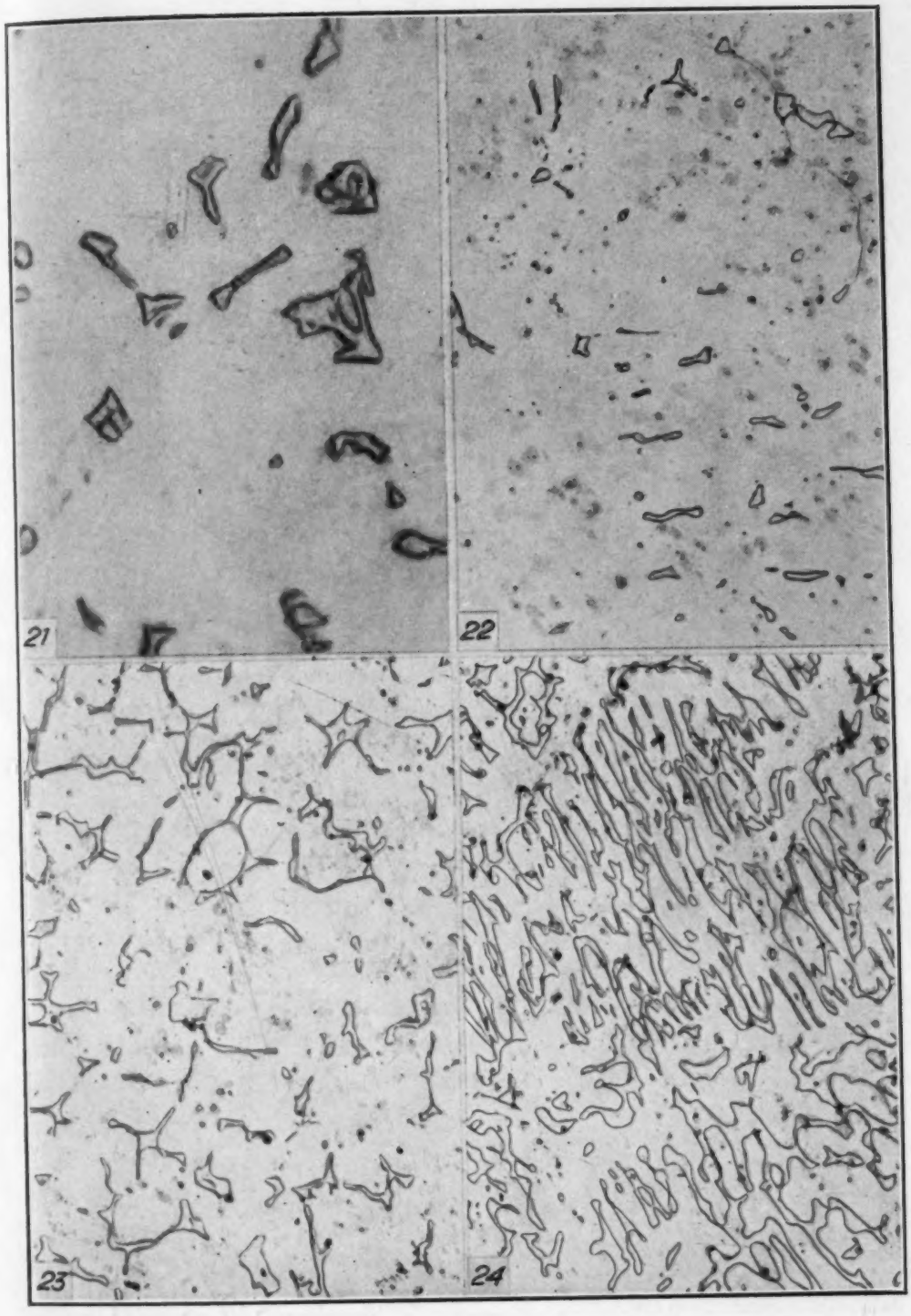

Fig. 21—Heat No. 13—Unetched. Titanium Carbide at × 1000.

Fig. 22—Heat No. 10, Etched. Alpha-delta Pools in an Austenitic Matrix, × 100.

Fig. 23—Heat No. 11, Etched. Alpha-delta Pools in an Austenitic Matrix, × 100.

Fig. 24—Heat No. 12, Etched. Alpha-delta Pools in an Austenitic Matrix, × 100.

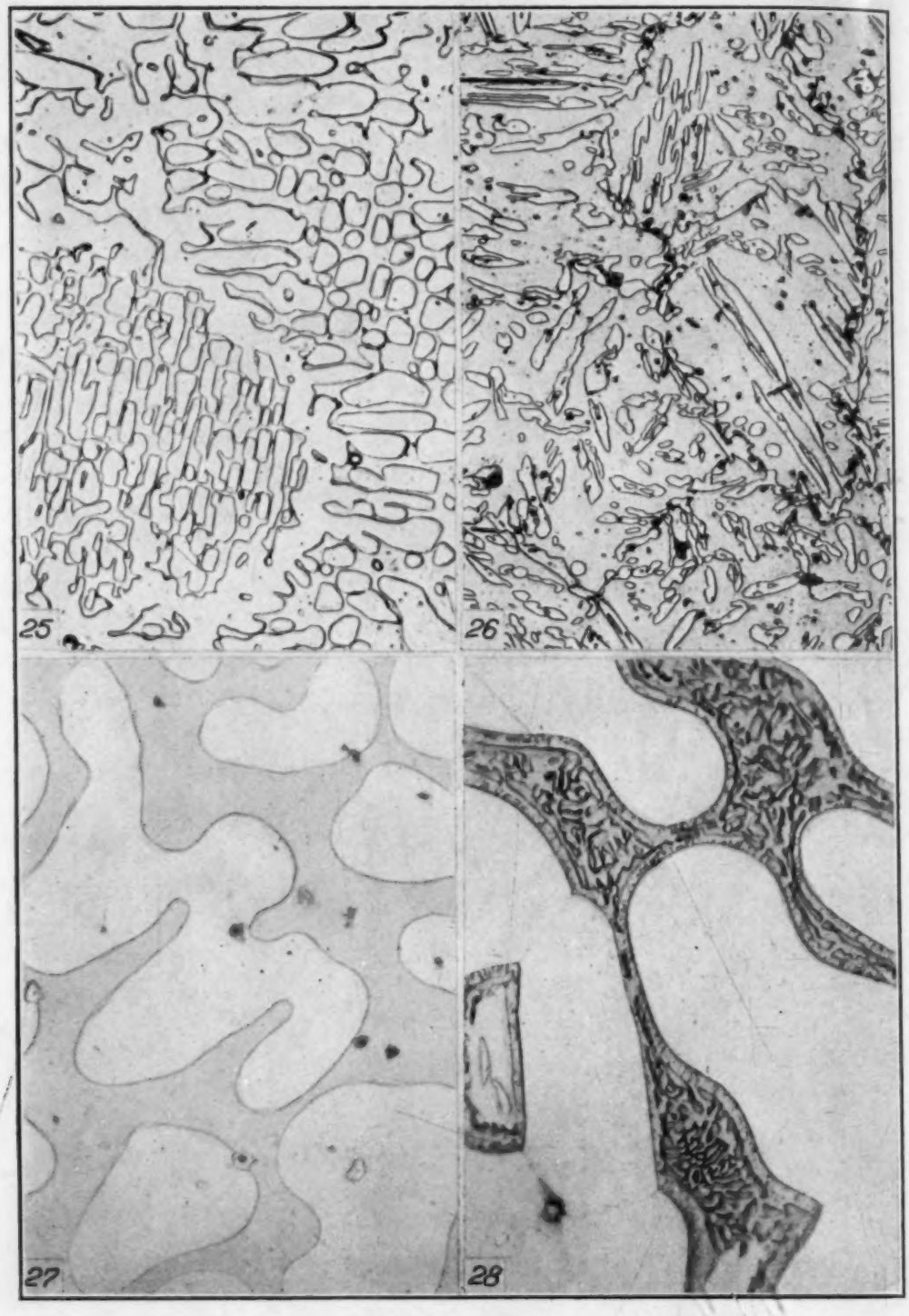

Fig. 25—Heat No. 13, Etched. Alpha-delta Pools in an Austenitic Matrix, × 100.

Fig. 26—Heat No. 15, Etched. Gamma Iron Grainlets in a Matrix of Alpha-delta Ferrite.

Fig. 27—Heat No. 13, Etched. Alpha-delta Pools in an Austenitic Matrix, × 500.

Fig. 28—Heat No. 13, Etched. After 8 Hours at 1250 Degrees Fahr. × 500.

Electrolytic etching in oxalic acid (14) uncovered further interesting information on the influence of titanium upon the microstructure. The most apparent, if not the most noteworthy, structural change induced by titanium is a partial suppression of the formation of austenite. Heat 10 which contains no titanium is almost completely austenitic, while in heat 15 which is high in titanium the truly predominating phase is alpha-delta solid solution. The action of titanium in altering the relationship between alpha and gamma iron is shown photomicrographically in Figs. 22 to 26. These views illustrate how increasing percentages of titanium augment the amount of chromium ferrite from 5 per cent in heat 10 to approximately 65 per cent in heat 15. The effect of titanium on the gamma iron loop is thus one which tends to compress or pull it in from the left to the right. Carbon is known to favor the formation of austenite (15), and therefore the extension of the gamma iron loop, but this loop-extending action of carbon is lost when it is combined with titanium. Then, too, titanium per se exerts a strongly suppressing effect on gamma iron formation. So by this double action of titanium the gamma iron loop can be practically eliminated.

All of the above steels showed a two-phase structure; all of them, too, exhibited a definite response to a magnet. The data in Table IX shows how magnetic susceptibility, alpha iron, titanium content, and density are correlated. Thus by simply noting the response an 18-8 steel shows to a magnet it is possible to predict not only the proportion of alpha and gamma iron in it but its tensile strength and titanium content as well.

Brief mention should be made of the effect of so-called sensitizing temperatures upon the structure of these titanium-treated

Table IX
Some Structural, Magnetic, and Corrosion Resisting Properties of 18-8 Titanium-Treated CRS Steels

Heat No.	Density gms./c.c.	Per Cent Alpha Iron*	Magnetic Attraction —Gms.	DuPont or Huey Acid Immersion Test	Condition after 2000 Hrs. in 4% Salt Spray
10	7.84	5	0.2	0.00093**	Perfect
11	7.80	25	1.2	0.00146	Perfect
12	7.74	50	5.0	.0.00202	Perfect
13	7.54	55	6.5	0.00149	Perfect
14	7.46	60	7.2	0.00300	Perfect
15	7.24	65	10.0	0.00784	Perfect

*Estimated from photomicrographs of Figs. 23 to 27 inclusive.
**Total inches penetration after three 48-hour periods.

steels. Specimens from each of the six heats under discussion, after having been exposed to a temperature of 1250 degrees Fahr. (675 degrees Cent.) for six hours, were etched electrolytically in a 10 per cent sodium cyanide solution and then most carefully searched under high magnification for newly precipitated phase material. No dark-etching precipitate or new constituent of any kind was discernible in heats 14 and 15. From this circumstance it may be inferred that all the carbon in them—at least all that in excess of the solubility limit (approximately 0.02 per cent)—had combined with titanium during the manufacturing and early solidification stages. In the ferrite pools of heats 10 to 13, however, there was found some eutectic-like material which was not formerly there. Its structure may be seen in Fig. 28. The chemical composition of the new component in that structure is not known. All these steels were subjected to the two most widely used corrosion tests, i.e., the 4 per cent salt spray and the Huey boiling nitric acid test. Reference to Table IX will show that titanium most emphatically does not lower the corrosion resistance of 18-8 to the salt spray. Macroscopic examination of the specimens after 2000 hours of continuous exposure to the salt spray failed to detect not alone any corrosion products but even the slightest staining. The corrosion resistance of these same alloys to boiling nitric acid, however, is inferior to low carbon titanium-free 18-8 steels. This is possibly due to the presence of alpha-delta ferrite which is believed to be less resistant to acid attack than gamma iron.

The age-hardening of austenitic steels by means of titanium has been investigated and reported by many researchers, notably Wasmuht and Kroll (16) in Germany and Hensel (17) in this country. They all seem to agree that about 3 per cent titanium is required for age-hardening. Heats 14 and 15, containing 0.90 and 3.50 per cent titanium each, were exposed to aging temperatures of 900, 1100, and 1300 degrees Fahr. (480, 595 and 705 degrees Cent.) for 8 hours. The hardness changes were as follows:

Treatment Degrees Fahr.	Brinell Hardness Heat No. 14	Heat No. 15
Quenched from 2000 degrees Fahr.	207	302
Quenched and aged at 900 degrees Fahr.	220	321
Quenched and aged at 1100 degrees Fahr.	248	415
Quenched and aged at 1300 degrees Fahr.*	235	395

*A pronounced loss in magnetism occurred at the above temperature.

It is generally acknowledged that austenitic corrosion resisting

steel is the most difficult of all to machine. According to Palmer (18), the factors which interfere with its machinability are: (a)—its toughness, (b)—its hardening under cold work and (c)—its high frictional characteristics. Titanium, which has an embrittling effect, mitigates toughness; and if absence of so-called "bugs" on the cutting edge of tools is a sign of low frictional properties, then it lessens frictional effect; it probably also, by promoting ferrite formation, tends to relieve the hardening due to cold working. At any rate, the writer has repeatedly observed—and his observations stand confirmed by machinists—that titanium-bearing 18-8 steels machine equally as well, if not slightly better, than either high sulphur or high phosphorus and selenium 18-8 steels. Heat 14 was machined with the usual machine shop tools at speeds ranging from 70 to 80 per cent of screw stock. All chips broke off short and clean.

The presence of small amounts of titanium in stainless steels has an enhancing effect upon their weldability. The writer has had occasion to examine microscopically a number of weld deposits laid with titanium-bearing 18-8 rods on titanium-bearing 18-8 base metal. He is not aware of ever having seen any abnormal or defective condition which was due to titanium. One interesting detail deserves attention: the structure of the weld metal is invariably one-phase, i.e., completely austenitic; that of the base metal is nearly always two-phase, or partly austenitic and partly ferritic. High percentages of titanium on the other hand are not conducive to welding quality. As we have seen they tend to make stainless steels ferritic, and ferritic steels are not easy to weld. The principal difficulty with welding ferritic stainless alloys arises from the fact that they are subject to rapid grain growth when heated to a high temperature and this grain cannot be refined by subsequent heat treatment. Such metals when welded develop a brittle zone immediately alongside the weld, and are apt to crack if deformed even slightly at room temperature.

Summing up, the results tabulated in Table VIII clearly indicate that titanium is a potent strengthening agent, increasing very greatly tensile and yield strengths. Concomitantly a definite, though not alarming, loss of valuable properties occurs, notably of impact strength and ductility. By producing alpha-delta iron in the microstructure of the low carbon stainless alloy titanium strongly inhibits susceptibility to intergranular corrosion. Unfortunately the obtainment of maximum quality in one direction is accompanied by a sacrifice of something in some other direction. Thus, the above

Table X
Chemical Composition and Physical Properties of Titanium-Treated Straight and Inverted Cast Monel Metals

Heat No.	Type of Monel	Chemical Composition Ni	Cu	Si	Fe	Mn	C	Ti
16	Straight	68.21	29.00	0.37	1.76	0.54	0.15	none
17	Straight	68.27	28.96	0.42	1.59	0.54	0.18	none
18	Straight	67.92	28.67	0.56	2.04	0.55	0.15	0.11
19	Straight	67.78	28.97	0.53	1.70	0.56	0.17	0.29
20	Straight	67.74	28.89	0.42	1.87	0.52	0.18	0.38
		Ni	Cu	Si	Fe	Mn	C	Zn* Ti
21	Inverted	30.30	67.93	0.30	0.89	0.43	0.14	1.00 none
22	Inverted	30.24	68.20	0.55	0.40	0.50	0.11	1.00 none
23	Inverted	28.80	68.82	0.62	0.93	0.57	0.11	1.50 0.15
24	Inverted	29.97	68.32	0.40	0.60	0.40	0.10	1.00 0.21
25	Inverted	36.50	60.19	0.66	1.43	1.02	0.12	1.25 0.08
26	Inverted	37.37	59.33	0.63	1.37	1.07	0.11	1.25 0.11

*The figures indicate the amount used in the charge. No trace of zinc was found in the specimens.

Heat No.	Type of Monel	Mechanical Properties (As-Cast) T. S. Lbs. Per Sq. In.	Y. P. Lbs. Per Sq. In.	Elon. %	Brinell Hardness	Izod Ft.-Lbs.
16	Straight	78,500	32,000	45.5	137	54.0
17	Straight	74,000	36,250	46.0	134	55.0
18	Straight	82,000	39,000	42.0	156	47.5
19	Straight	88,000	42,000	42.5	170	47.0
20	Straight	91,250	42,250	40.0	179	43.0
21	Inverted	59,500	28,250	34.5	131	36.0
22	Inverted	61,750	30,500	31.5	137	32.5
23	Inverted	69,000	44,500	24.5	156	30.0
24	Inverted	75,000	49,500	23.0	163	22.5
25	Inverted	72,750	37,500	40.0	143	34.5
26	Inverted	75,250	53,000	45.5	163	38.0

advantage is offset by impairment of welding quality and corrosion resistance in nitric acid. Unlike other embrittling elements which are used to make metal machinable, titanium aids machinability without lowering the resistance of the metal to corrosion by salt spray.

Straight (30-70) and Inverted (70-30) Monel Metals

In Table X there are shown eleven different melts of copper-nickel alloy; the first five are straight monel metals; the last six are also monel metals but of the inverted or 70-30 type. Each group, in turn, consists of titanium-treated and titanium-free metals. These eleven heats were selected at random from among scores of others of almost identical composition which had been manufactured in the course of regular production. Their properties, therefore, are representative of the group to which they belong.

The best cast monels rarely pull in excess of 75,000 pounds per square inch and conjunctively stretch to an elongation of 35 per cent. Some nonferrous foundries have difficulty in obtaining from their

copper-nickel alloys as much as 65,000 pounds tensile strength and 25 per cent elongation, which, according to Navy Department Specifications 46Mld, are the minimum requirements for this material. The addition of a small amount of low carbon ferrotitanium to monel metal will give it a remarkably high combination of ductility and

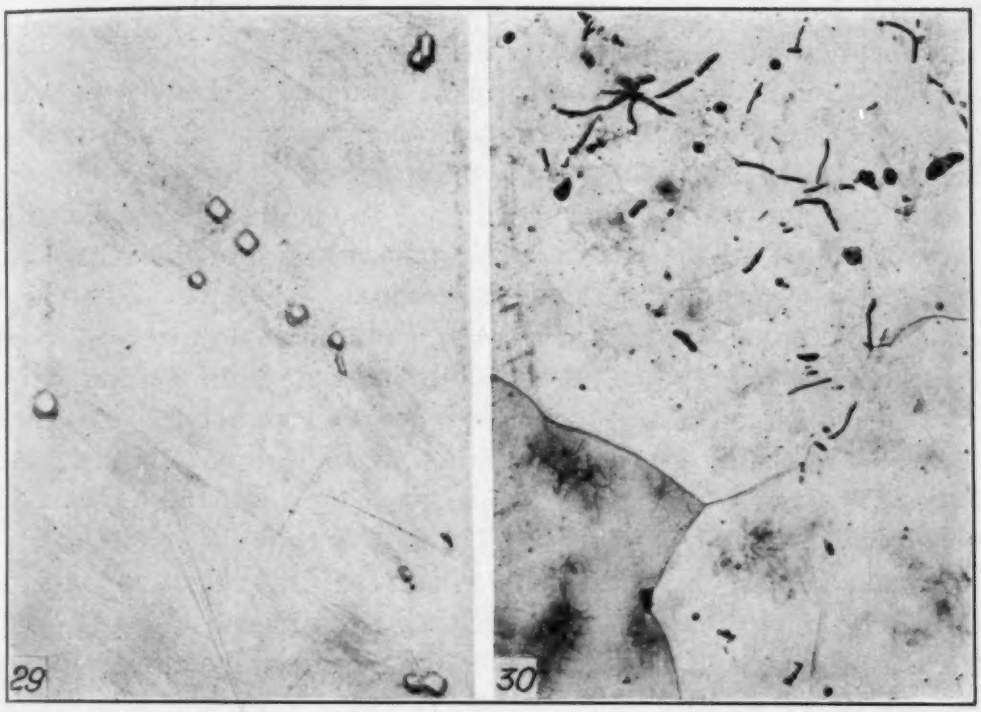

Fig. 29—Heat 24, Unetched. Note the Absence of Graphite. × 100.
Fig. 30—Heat 21, Etched. Note the Presence of Graphite. × 100.

strength. The test data recorded opposite monel heats Nos. 18, 19, and 20 in Table X show what properties may be expected from this material when treated with titanium.

Titanium has a similar good effect on inverted monel. This is a very finicky alloy, sensitive to the slightest variations in composition, giving—if not "balanced" properly with respect to silicon, iron, and manganese—either very high values of tensile strength and low ductility, or the converse, i.e., low strength and high ductility. Copper-nickel compositions which have high initial ductility and moderate tensile strength can have their strength stepped up appreciably without too great a loss of ductility if treated with titanium. Heats Nos. 23 to 26 bear witness to this fact. Heats Nos. 21 and 22 are not very good comparison heats. Had heat No. 21 a higher silicon and manganese content (0.60 per cent and 0.70 per cent respectively), and

heat No. 22 a higher iron content (1½ times the silicon or 0.85 per cent), they both would have given higher tensile properties. It should be noted that heats Nos. 25 and 26 are similar to each other, but differ with respect to the amount of copper and nickel they contain from heats Nos. 23 and 24, which also are similar to each other.

The solubility limit of carbon in cast 70-30 copper-nickel alloy of normal silicon content (0.40 per cent to 0.70 per cent) is at the very most approximately 0.04 per cent carbon. If present in excess of this amount it will occur in the form of graphite carbon segregations such as shown in Fig. 30. Microscopic examination of titanium-treated monel metals will disclose the presence of carbon in the combined and not the graphitic condition. In both ferrous and non-ferrous alloys the carbide of titanium is identically the same as to crystal form and metallographic appearance. The increased strength and almost unimpaired ductility of titanium-treated monels is due in no small part to their greater freedom from graphitic fissures.

One other advantage which titanium-treated monels have over untreated graphitic monels is superior machinability. The graphite flakes in titanium-free monels are too few, too thin, and too small to be of any benefit from the standpoint of machining. The carbide of titanium, due to its hardening and slightly embrittling effects, causes the chip to break off more cleanly and in smaller fragments.

Recently, O. B. J. Frazer (19) announced the discovery and the immediate addition to the family of monel metals of a new free-machining "R" monel. Sulphur is the element which makes this alloy machinable. Whenever elements like lead, sulphur, or selenium are incorporated in the analysis for the obtainment of greater machinability it nearly always is necessary to accept reductions in tensile strength and yield strength of as much as 15 to 20 per cent from the values for the corresponding ordinary alloys. Other properties, to mention only two, corrosion and weldability, also are impaired. Titanium, while not making the alloy a strictly free-machining one, does improve to an appreciable extent machining quality, without a sacrifice of any but the impact properties of the metal.

Summary

A summary of some of the more important results of this work follows.

1. Titanium, when added to low carbon irons and medium

carbon steels in amounts under 0.20 per cent, increases tensile strength, yield point, and hardness with only a slight diminution of ductility and notch-impact resistance. It enhances general quality, for titanium-treated steels are low in nitrogen content, homogeneous in structure, free from blowholes, and contain a minimum of oxidized slag particles. Additions from 0.20 to 0.60 per cent embrittle the steel both dynamically and statically; steels alloyed with more than 0.60 per cent titanium, though ductile statically, show abnormal brittleness when notched. The structure of high titanium steels, under all conditions of treatment, is one which consists of coarse crystals of insoluble titanium carbide embedded in a matrix of soft ferrite.

2. Titanium additions to nickel-chromium steels raise tensile and yield strength; this increase is continuous up to approximately 0.40 per cent titanium, while the ductility is only slightly affected up to about 0.20 per cent titanium, above which it sharply falls. Excessive additions of titanium (over 0.60 per cent) aside from lowering the tensile strength to a certain degree, exert the maximum effect by a serious decrease in the impact strength of the steel. Nickel-chromium steel containing 1.84 per cent titanium is free from precipitation hardening effects; its structure consists of titanium carbide segregated in the boundaries of polygonal ferrite grains.

3. Titanium has little advantageous influence upon the properties of copper-bearing steel. The presence of the element raises its tensile strength and at the same time reduces greatly its static and dynamic ductility. It also tends to decrease the solubility of copper in the solid solution.

4. Titanium exerts a definite alloying effect in cast 18-8 stainless steel, materially raising tensile strength, yield point, and hardness; this increase is continuous up to 3.50 per cent titanium; static ductility is lowered gradually up to about 0.60 per cent titanium, more rapidly from 0.60 to 1.00 per cent titanium, and at 3.50 per cent it is practically nonexistent; dynamic ductility is affected similarly, the high titanium specimens breaking off sharply instead of deforming in the "taffy candy" manner characteristic of tough austenites. It promotes the formation of magnetic alpha-delta ferrite. Magnetic tests are capable of furnishing a positive criterion of tensile properties. Titanium strongly inhibits susceptibility to intergranular corrosion; while it does not decrease resistance to corrosion in sea salt spray, it does lower the resistance of the metal to attack

by nitric acid. The machining and welding qualities of the material are greatly enhanced.

5. Titanium is truly beneficial to copper-nickel alloys, and deserves to be added to the list of useful alloying elements for cast monel. Resistance to corrosion in salt atmosphere is not reduced; machinability and tensile properties are improved.

Acknowledgments

This investigation was carried out at the suggestion of T. N. Armstrong, formerly assistant metallurgist, Norfolk Navy Yard, now associated with the International Nickel Co., whose encouragement and active co-operation made the study possible. Thanks are due also to F. B. Anderson, melter, Norfolk Navy Yard, for the preparation of the various alloys.

The writer wishes to express his appreciation to Dr. G. F. Comstock of the Titanium Alloy Manufacturing Company for his help in securing the analysis mentioned on page 790.

Bibliography

1. Bradley Stoughton, "Railway Age Gazette," Feb. 7, 1913.
2. F. M. Becket and R. Franks, "Titanium and Columbium in Plain High-chromium Steels," *Transactions*, American Institute of Mining and Metallurgical Engineers, Vol. 113, Oct. 1933.
3. Strauss, Houdremont and Their Associates, *Stahl und Eisen*, 1930, p. 1473 and 1526.
4. E. C. Bain, R. H. Aborn, and J. J. B. Rutherford, "The Nature and Prevention of Intergranular Corrosion in Austenitic Stainless Steels," Transactions, American Society for Steel Treating, Vol. 21, 1933, p. 481.
5. L. B. Bliss, "Welding Rod Coatings," Foote-Prints, June, 1934. A publication of Foote-Mineral Co.
6. Patentees: T. N. Armstrong and F. B. Anderson. Patent Pending.
7. T. J. Cunningham, "Determination of Titanium in Plain Carbon and 18-8 Nickel-Chromium Steels," *Industrial and Engineering Chemistry*, Vol. 5, No. 5, p. 305-6, Sept. 15, 1933. Analytical Edition.
8. William M. Thornton, Jr., "Titanium," first edition, 1927, p. 52, The Chemical Catalogue Company.
9. T. N. Armstrong, "Properties of Some Cast Alloy Steels," Transactions, American Society for Metals, Vol. 23, p. 286, 1935.
10. K. S. Seljesater and B. A. Rogers, "Magnetic and Mechanical Hardness of Dispersion Hardened Iron Alloys," Transactions, American Society for Steel Treating, Vol. 19, p. 553, 1932.
11. G. F. Comstock, "New Role of Titanium in Steels and Alloys," Metal Progress, Jan. 1935, p. 37.
12. J. L. Gregg and B. N. Daniloff, "Alloys of Iron and Copper," First Edition, McGraw-Hill Book Co., Inc., p. 186.
13. The Book of Stainless Steels, published by The American Society for Steel Treating, Cleveland, Ohio, Chapter 14, p. 323-354.
14. G. A. Ellinger, "Oxalic Acid as An Electrolytic Etching Reagent for Stainless Steels," Transactions, American Society for Metals, Vol. XXIV, No. 1, March 1936, p. 26-35.

15. V. N. Krivobok and M. A. Grossmann, "A Study of the Iron-Chromium-Carbon Constitutional Diagram," TRANSACTIONS, American Society for Steel Treating, Vol. 18, p. 760, 1930.
16. Washmuht and Kroll, *Journal,* British Iron and Steel Institute, 1931, II, p. 670.
17. F. R. Hensel, "Age-Hardening of Austenite," *Transactions,* American Institute of Mining and Metallurgical Engineers, Iron and Steel Division, 1931, p. 255.
18. The Book of Stainless Steels, American Society for Metals, Chapter VI, p. 98.
19. O. B. J. Frazer, "A Free Machining Monel Metal," *Iron Age,* August 13, 1936.

DISCUSSION

Written Discussion: By G. F. Comstock, Titanium Alloy Mfg. Co., Niagara Falls, N. Y.

We are very glad indeed to see this paper on titanium steels presented for discussion and to note the careful work that the author has carried out in testing them.

The description of the new method of producing such steels is however rather disappointing, since the slag used, which evidently is the critical part of the process, is not described. We feel that the author has been perhaps a trifle over-optimistic in his enthusiasm for the new process, and frankly we doubt that the reaction between aluminum and the various forms of titanium oxide, especially "titanalba," was as complete as claimed. Our experience has been that pure white titanium oxide is less easily reduced than rutile which contains iron oxide, and the analyses which we made for the author were only those that he reports for the rutile-treated heats. The analyses he mentions on the products of the titanalba reactions were evidently made elsewhere, and we doubt therefore whether the comparison of the two methods is justified on the basis of analytical results made probably by different methods and certainly in different laboratories. We believe that much lower aluminum contents can be obtained in titanium alloy steels if a good grade of low-aluminum ferrotitanium is used as the source of titanium, instead of using metallic aluminum and titanic oxide as the author has done.

The presence of Fe_3Ti in eutectic form in Heat No. 1 is surprising; possibly this is some other compound due to nickel or silicon in the steel, or is due to dendritic segregation. Age hardening would not be expected without more titanium, in pure iron-titanium alloys, and it would also of course be expected that in such alloys with only 1.26 per cent titanium all the titanium should remain dissolved.

What the author calls titanium carbide we usually refer to as cyanonitride, as it often seems to be colored more like the latter compound. The author's remarks regarding it agree in general with our experience, but it might be added that as shown by Mr. Bannon in another paper presented before this convention, it appears to be soluble in steel above 2250 degrees Fahr. This is about the upper limit for rolling temperatures, and the solubility at such temperatures, as well as distortion in working, probably enters into their improved distribution in wrought steels. The remark regarding grain size at the top

of page 800 would be more appropriate if it were confined to "cast steel" instead of "steel" in general.

The author's statement regarding nitrogen under item 2 on page 800 is very interesting. Does he have any data to offer in support of it?

There seems to be some mistake on page 801, either in the tables or in the text, for they do not support each other. No data on additions of less than 0.2 per cent titanium to the nickel-chromium steel are presented in support of the conclusion that such additions are beneficial. Could the author in his reply to the discussion, correct this omission and supply the data on which he based the conclusion stated in the first sentence of the second paragraph on page 805, and again in the second paragraph on page 817?

The data on titanium in stainless steel are valuable and timely. The effects on mechanical properties which are brought out here have also been noted in somewhat less detail by Bennek and Schafmeister in a paper entitled "Precipitation Hardening of 18 Per Cent Chromium 8 Per Cent Nickel Steels by Means of Beryllium, Boron, or Titanium," published in 1932 in Archiv. f.d. Eisenhuettenwesen, Vol. 5, p. 615. This reference might well be added to the author's bibliography.

In the discussion of the effects of titanium on the microstructures of these highly alloyed steels, more attention might have been paid to variations in silicon, chromium and nickel contents between the titanium and non-titanium steels, though the author's observations regarding the effect of titanium are probably correct in a general way. The improved machinability due to titanium is a most interesting observation which does not seem to have been reported previously. The results with titanium in monel metal also seem to be new and most important.

Written Discussion: By Russell Franks, research laboratories, Union Carbide and Carbon Research Laboratories, Inc., Niagara Falls, N. Y.

Mr. Duma's paper relating to the effects of titanium on cast metals contains considerable information that should serve as a guide for those contemplating the use of this element in castings of various metals. Our laboratories have conducted a relatively large number of tests involving the addition of titanium and other strong carbide-forming elements to low and high alloy steels, and as shown in this paper elements of this type produce a marked effect on the physical characteristics of both cast and wrought steels. We fully agree that the location of the carbides or other compounds formed as a result of the titanium addition to cast steels is responsible for the impairment of toughness even though the hardening ability of the metal is greatly reduced. These carbides or other compounds segregate in the grain boundaries of the cast steel and greatly reduce toughness, and in most instances this is associated with an increase in grain size. These effects are most pronounced when the cast steels contain at least enough titanium to combine with the carbon present. Ordinarily this means that the titanium content is four times the carbon content, although when greater amounts of titanium are incorporated the result is to render the cast steel even less tough.

We also fully confirm his findings, which show that smaller amounts of titanium produce a definite improvement in the quality of low and medium carbon steel castings, and we might add that small additions of zirconium like-

wise enhance ductility and reduce the hardness of castings of this type without seriously affecting other desirable properties. Whether this is due to the fact that the titanium or zirconium combines with the nitrides or removes the evil effects of another impurity is a question that can hardly be answered. However, in both instances the result is to enhance the properties of the castings rather than detract from them.

Although Mr. Duma states that his observations in regard to the presence of appreciable percentages of titanium pertain to castings rather than to wrought products, we believe that this point should be emphasized to a greater degree because depending on the relation of carbon to titanium the results obtained in wrought products are vastly different from those secured in castings. It has been definitely established that the addition of judicious amounts of titanium to the 4-6 per cent chromium steels reduces their air hardening capacity. For instance a cast steel containing 6.43 per cent chromium, 0.11 per cent carbon, and 0.48 per cent titanium was tested for strength, ductility, toughness and hardness, both in the cast condition and after annealing according to a number of different procedures. The results showed that the cast steel was substantially free of air hardening, had good strength and was sufficiently ductile for most purposes, although the highest Izod impact value obtained was in the neighborhood of 5 foot-pounds, showing that the resistance to shock was low. The investigation of a forged and rolled steel that contained 6.46 per cent chromium, 0.08 per cent carbon, and 0.42 per cent titanium gave even in the as hot-rolled condition an impact value of 20 foot-pounds, whereas after annealing at 750 degrees Cent. the same steel exhibited an Izod impact value of 89 foot-pounds, revealing that in addition to the softness and ductility possessed by the metal, it was extremely tough. Our only explanation for the difference in the behavior of titanium in the cast and wrought steels is that the particles occurring in the grain boundaries of the cast steels are distributed at random throughout the steels during the hot working operations with the result that toughness of the metal is not detrimentally affected. However, this result cannot be obtained even in wrought 4-6 per cent chromium steels when more than seven times as much titanium as carbon is present.

In the discussion of the effects of titanium on cast 18 per cent chromium and 8 per cent nickel steels, Mr. Duma indicates that the titanium addition is the only practical method of securing in these steels immunity from intergranular attack, and mentions that while other elements such as columbium, tantalum, and uranium can be employed, the use of these alternate remedies is not always applicatory nor are any of them comparable to titanium in lowness of cost. We cannot agree with this statement. The columbium addition can be used in castings of these steels to enhance their resistance to intergranular attack and we are unaware of a single instance where the use of columbium cannot be applied because of cost deferential or for any other reason. The facts are that by the use of columbium certain difficulties that are encountered in using titanium are eliminated. The addition of titanium increases the sluggishness of the molten austenitic chromium-nickel steels and in some instances difficulty is met with in securing castings of a high quality, while in the case of columbium, the presence of this element does not change the pouring characteristics of molten 18-8 steel, and castings of high quality are obtained.

Furthermore, 80-90 per cent of the columbium introduced into the melt can be recovered in the finished product but in the case of titanium a recovery in the vicinity of 50-60 per cent is all that can be expected. If we take the cost of the titanium addition against the cost of the columbium addition it will be found that there is comparatively little difference in the cost of these two elements in the finished steel, and even though the titanium addition might have a slight advantage as far as initial cost is concerned, this is offset by the fact that columbium-bearing steel scrap can be remelted with a relatively high recovery of columbium in the finished product. The columbium-bearing 18-8 steels can be welded without serious loss of columbium in the weld metal provided the proper technique is employed, although it is generally recognized that titanium cannot be retained during the welding operation. In fact, the safest procedure at present is one that consists of using a columbium-bearing 18-8 steel rod when it becomes necessary to weld the titanium-bearing 18-8 steels because sufficient columbium can be recovered to impart to the welded structure comparative freedom from intergranular attack. As in the case of titanium, the columbium addition must be controlled so as to obtain a steel having optimum physical characteristics associated with the necessary resistance to intergranular attack, and the amounts of columbium required for this purpose have been reported elsewhere in the literature.

Written Discussion: By J. J. Curran, Walworth Co., Greenburg, Pa.

This paper, and some of the discussion from the floor, indicates clearly that the effects of various elements in alloys may depend greatly on the condition in which the alloy is used or tested, and even on the method by which it is manufactured.

For instance, we know that the use of a slight excess of aluminum as a final deoxidizer promotes fine grain in wrought steels. With this fine grain is usually associated a maximum of toughness and ductility. However, in the case of acid electric steel castings, the use of this same slight excess of aluminum causes segregation of the solid nonmetallic impurities to the grain boundaries, resulting in low ductility and toughness. The networks of inclusions in the grain boundaries are not affected by heat treatment, hence the "effective grain size" of the casting after heat treatment may equal that of the untreated casting.

In the case of basic steel castings, this condition is less noticeable, due to the generally lower sulphur contents of the basic steels. In these steels, the aluminum tends to cause formation of grain boundary networks, but due to the smaller quantities of sulphides available, the networks are not nearly so complete or extensive, and less lowering of ductility results.

Titanium, in the basic cast steels described in the paper, apparently produces a similarly weak network of particles of a titanium compound around the individual grains. These particles are not affected by heat treatment, hence the castings are characterized by low ductility and toughness, coincident with a large "effective grain size." The same steels, however, in the wrought condition, would doubtless exhibit highly satisfactory toughness, due to destruction of continuity of the inclusion networks in the hot working operations.

The author's references to the erratic behavior of the 70-30 copper nickel alloy, and the importance of "balance" between the minor elements, iron, man-

ganese, and silicon, are very interesting. It is unfortunate that he did not devote more space to this subject, and give in some detail the methods of controlling the physical properties of this "finicky" alloy.

Author's Closure

The author appreciates very much the discussions which have been submitted in connection with this paper. The findings which led the author to state that steels treated with titanalba are more likely to contain less residual aluminum than those treated with rutile were the following:

Three 1000-pound heats of 18-8 stainless steel were prepared for study, each similar to the other in all manufacturing details but one, i.e., the manner in which the titanium additions had been effected. The charge for each heat consisted of approximately 1000 pounds of cold-rolled steel plate scrap, 20 pounds nickel oxide, 20 pounds lime, and 15 pounds fluorspar. When each respective charge had melted, additional lime and fluorspar were added. This first slag was then removed and replaced with one consisting entirely of lime. After the second slag had become liquid, it was thinned and brought to the required consistency with further occasional additions of lime mixed with aluminum shot. Ferrosilicon and ferrochromium were next introduced to the metal. A few minutes prior to tapping, the first heat was treated with approximately 12 pounds of 35 per cent ferrotitanium; the second, with 10 pounds rutile and 5 pounds aluminum; and the third, with 10 pounds titanalba and 5 pounds aluminum. Chemical analysis found the following percentages of titanium and aluminum present in each heat.

Heat	Per Cent Titanium	Per Cent Aluminum
First	0.28	0.11
Second	0.35	0.06
Third	0.32	0.03

The per cent recovery of titanium and the residual aluminum vary considerably with the type of slag, the composition of the bath, and the amount of titanium added to the bath. For instance, it has been found that the highest recovery of titanium for monel metals is obtained when aluminum oxide is the slag and titanalba together with aluminum are the source of titanium; for stainless steel, when lime and aluminum are the slag materials and rutile and aluminum the source of titanium; for medium carbon and low alloy steels, when the slag is calcium-alumina-carbide and ferrotitanium the source of titanium. With the thermit process, additions from 1.00 to 2.00 per cent titanium can be effected with a recovery of 80 to 90 per cent; additions from 0.50 to 1.00 per cent titanium can be obtained with a recovery approximating 70 per cent; additions of titanium under 0.50 per cent are obtainable with a recovery of 55 to 60 per cent. All these percentages can be bettered slightly if the amount of aluminum is increased to a figure in excess of that theoretically required to reduce the dioxide of titanium. At present, all monel metal heats, all stainless steel heats, and all straight carbon and low alloy steel heats made in the Yard are treated with titanium, the source of titanium for each being titanalba, rutile, and ferrotitanium respectively.

The author chose to call the metallic particles shown in the photomicrographs titanium carbide rather than titanium cyanonitride because, when examined in white light on ground glass none of them exhibited any of that pink color which identifies cyanonitride. Their color was brilliantly metallic; their form angular and irregular; polishing did not cause them to pit. Moreover, their number increased as the amount of pearlite in fully annealed steel samples decreased, a circumstance which—in the absence of evidence of the more direct type—finally decided their name.

The assertions made concerning titanium additions under 0.20 per cent as affecting favorably the tensile properties of nickel-chromium steels are based not entirely on the data presented in the Tables but on hundreds of production heats of nickel-chromium-molybdenum and nickel-chromium steels of slightly different composition. In order to bring out more clearly the influence of titanium on nickel-chromium steel the author used only such heats as were nearly alike to each other in chemical composition as possible. Unfortunately he did not have a low titanium nickel-chromium heat approximating the composition of those listed in the Tables. For several years past the Yard has been using titanium as a final addition to all its steels and incorporating with them from 0.05 to 0.20 per cent titanium. Whenever the titanium treatment was omitted trouble was generally experienced either in meeting the tensile requirements of the specifications or with internal unsoundness and porosity. On the other hand, if there were more than 0.20 per cent titanium in the metal it also generally failed, either with respect to elongation because of the consequent embrittlement, or with respect to cold bending. For example, chain steel, the chemical composition of which is—

C	Mn	P	S	Si	Ni	Cr	Mo	Ti
0.25/0.35	0.70/0.85	0.04	0.04	0.10/0.30	1.40/1.60	0.50/0.80	0.20/0.30	0.20 Max.

and the physical requirements for which are—

Tensile Strength Pounds Per Square Inch	Yield Point Pounds Per Square Inch	Elongation Per Cent	Reduction of Area Per Cent
125,000	93,750	12	20

gives, when treated with 0.05 to 0.20 per cent titanium, values ranging from—

Tensile Strength Pounds Per Square Inch	Yield Point Pounds Per Square Inch	Elongation Per Cent	Reduction of Area Per Cent
135 to 150,000	120 to 135,000	12 to 16	28 to 38

If no titanium is used the resulting tensile strength will average about 125,000 pounds per square inch; if more than 0.20 per cent titanium is added the resulting elongation will average under 12 per cent.

Concerning nitrogen content in titanium bearing steels, the following three heats were analyzed for nitrogen, and with the following results:

Heat No.	Per Cent Titanium	Per Cent Nitrogen
3	0.18	0.003
4	0.20	0.005
5	1.05	0.003

In the discussion of the effects of titanium on the microstructure of 18-8 stainless steels, Dr. Comstock states that the author might have paid more attention to variations in silicon, chromium, and nickel contents. This certainly was done. Every analysis was taken apart piecemeal and attempts were made to correlate these variables with both microstructure and physical properties. The attempts were unsuccessful.

Mr. Russell Franks' remarks concerning the many advantages possessed by columbium over titanium as a stabilizing element in 18-8 stainless steels, are well taken and are concurred in. When the author said that other stabilizing elements were not comparable to titanium in lowness of cost he meant initial cost, and that of the dioxide of titanium.

The comments of Mr. Curran are very welcome. The subject of "balance" and the influence of the various elements commonly found in 70-30 copper-nickel alloys will be treated separately and at greater length in another paper which the author hopes soon to complete.

THE BEHAVIOR OF SOME LOW ALLOY STEELS IN THE SINGLE-BLOW DROP TEST

By Owen W. Ellis

Abstract

The percentage reduction in height in the single-blow drop test at forging temperatures varying from 600 to 1200 degrees Cent. (1110 - 2190 degrees Fahr.) of normal 1-inch samples cut from a series of steels has been measured. Graphs are presented showing the relationship between percentage deformation and temperature.

Reactions to forging at various temperatures of two steels of similar carbon content, but of different inherent grain size, are considered. It is concluded that the grain size at the forging temperature of steels of low and medium carbon content has a measurable effect upon their resistance to deformation.

Comparisons are made between the behavior of three low alloy steels (Cor-Ten, Man-Ten, and Sil-Ten) and three straight carbon steels of similar carbon content. A comparison is also made between a medium manganese steel (Man-Ten) and a straight carbon steel of approximately equal tensile strength at room temperature.

Introduction

In A previous paper[1] the author described the results of a number of experiments on the forgeability of steel. The methods of test, as well as the effects on forgeability of carbon, nickel, chromium, nickel plus chromium, and chromium plus vanadium, were considered.

Carbon was found to have a predominating effect upon the forgeability of steel. The addition of nickel, of chromium, and of nickel plus chromium to low carbon steels appeared to have but slight influence upon their forgeability; on the other hand, the addition of these elements to medium carbon steels reduced their forgeability—possibly more so at high than at low temperatures. The effect of vanadium on otherwise similar steels was shown in Fig. 17 of the previous paper (here reproduced as Fig. 1).

[1]O. W. Ellis, "Further Experiments on the Forgeability of Steel," Transactions, American Society for Metals, Vol. 21, 1935, p. 673.

A paper presented before the Eighteenth Annual Convention of the Society, held in Cleveland, October 19 to 23, 1936. The author, Owen W. Ellis, is Director of Metallurgical Research, Ontario Research Foundation, Toronto, Canada. Manuscript received April 23, 1936.

The effects upon forgeability of variations in melting practice and in composition of the charge of eutectoid steels of approximately the same analysis were also described.

Description of Present Experiments

With the co-operation of the Carnegie-Illinois Steel Corp., through the American Sheet and Tin Plate Co., the author has been enabled to measure the reactions to forging at various temperatures of two steels of similar carbon content but of different inherent grain size, and to compare the forgeabilities of three low alloy steels

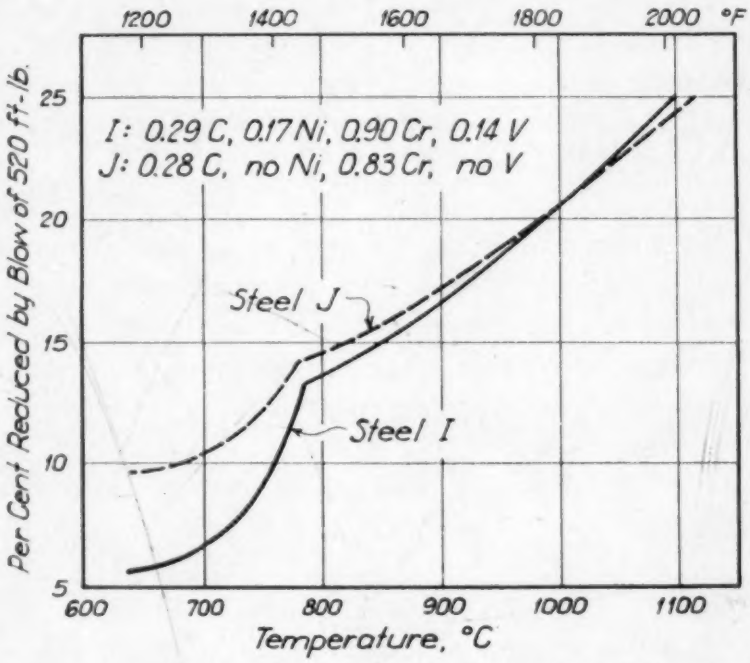

Fig. 1—Effect of Vanadium on the Forgeability of Chromium Steels.

(Cor-Ten, Man-Ten, and Sil-Ten) with those of three commercial carbon steels of similar carbon content. A comparison has also been made between Man-Ten and a carbon steel of approximately the same tensile strength.

The present experiments were conducted on test samples 1 inch high and 1 inch in diameter. The blows were applied to the samples by means of a hammer weighing 113 pounds. This hammer was allowed to fall from a height of 55½ inches to the top of the test samples. The energy of the blow given the samples was therefore 520 foot-pounds.

The samples were heated to the various forging temperatures in an electric resistor furnace, which could be controlled within relatively close limits by means of a pyrometer controller. This controller was set in such a way as to hold the furnace at a constant temperature. When it was at a constant temperature, two samples were then placed within the furnace. One of these was drilled to a point a little over half way through. Into the hole thus produced the hot junction of a pyrometer was snugly fitted so that the temperature of the sample could be observed by suitable means outside the furnace. A portable potentiometer was used for this purpose. The other sample was placed on top of the first, and was removed from the furnace for forging after it had remained in position for 25 minutes. Just before it was removed from the furnace, an observation was taken of the temperature of the lower sample. It was assumed that both samples were at the same temperature, and that the upper sample was therefore forged at this temperature.

In due course, other samples were substituted for the second sample, the temperature of the furnace being altered to suit the requirements of the experiments, and measurements being taken of the temperature of the lower sample as in the first instance. At regular intervals the pyrometer used to determine the temperature of the lower sample was checked to ensure that correct observations of forging temperatures were being made.

After the forged samples had cooled to room temperature, their heights were measured. The initial heights being known, the heights after forging were subtracted from the initial heights and then divided by the initial heights in order to obtain (by multiplying these values by 100) the percentage reductions in height of the samples.

Criticisms of Single-Blow Drop Tests

The question may be raised whether the results of tests in single-blow drop-test machines afford a complete measure of forgeability.

In this connection it may be observed that considerable difference of opinion exists as to the meaning of the word "forgeability." The author prefers the word "malleability" to express that property, or group of properties, which is measured by the single-blow drop test, and, in presenting the results of his work to other societies, has invariably used the term "malleability." It is of interest, however, to record that, whenever the question has been raised as to what term

should be used in presenting the results of his work to groups in the United States, the word "forgeability" has invariably been chosen by editors in preference to the word "malleability."

The single-blow drop test is recognized by the author as indicating only the resistance to plastic deformation of samples of steel at the specified temperatures used in forging. It gives no indication whatever of the readiness with which steel can flow in closed dies, nor of the character of the steel subsequent to such an operation as drop forging.

It has been pointed out on many occasions that forgers in the trade, who manufacture a wide variety of sections—from most intricate small tools, such as scissors and wrenches, to large sections, like crank-shaft blanks—are almost uniformly of the opinion that fine-grained steel is more plastic and therefore fills dies of intricate sections better than coarse-grained steel. As a result, both production and die life are increased. In addition to these advantages, fine-grained steel is less susceptible to flash cracks in both hot and cold trimming. Certain defects, such as worm holes, disclosed in heat treated and pickled forgings, are the results of highly localized stresses but are practically unknown in sections difficult to forge when fine-grained steels are used.

The single-blow drop test, when cylindrical test samples are used, gives scarcely any indication of the susceptibility of steels to such defects as flash cracks. It is not improbable, however, that forms of test sample might be devised which would enable the objectionable characteristics of certain steels to be brought out quite clearly.

The writer is of the opinion that it cannot be said that the single-blow drop test fails entirely to indicate the degree to which a steel might flow in closed dies. In this connection it is of interest to note the results recorded in the following section on the influence of actual grain size on the forgeability of steel, and particularly to observe the relative behaviors at high temperatures of steels which are inherently fine-grained and coarse-grained, respectively.

Influence of Austenite Grain Size on the Forgeability of Steel

To measure the effect of austenite grain size upon the forgeability of steel, two steels of like analysis but of different inherent grain

size were tested. These steels were of the following compositions and mechanical properties.

	"Coarse-grained" Steel A	"Fine-grained" Steel B
Carbon	0.42 per cent	0.41 per cent
Manganese	0.74 per cent	0.70 per cent
Sulphur	0.028 per cent	0.022 per cent
Phosphorus	0.020 per cent	0.016 per cent
Silicon	0.24 per cent	0.23 per cent
Limit of proportionality (pounds per square inch)	22,500*	17,500
Yield point (pounds per square inch)	50,500*	47,875*
Tensile strength (pounds per square inch)	90,675*	85,750*
Elongation (per cent on 2 inches)	28¾*	25*
Reduction of area (per cent)	54½*	54½*

(*Mean of two tests.)

The forgeability-temperature curves for these steels, given in Fig. 2, show that the "coarse-grained" Steel A was softer than the "fine-grained" Steel B at all temperatures up to about 975 degrees Cent. (1790 degrees Fahr.), but harder at temperatures above that point. Microscopic examination showed clearly that at some temperature intermediate between A_1 and 1200 degrees Cent. (2192 degrees Fahr.) the austenite grain sizes of these steels were transposed. Figs. 3 to 8 show the structures of samples of these steels, which had been heated in pairs to three different temperatures.

Figs. 3 and 4 show the structure of samples which had been heated to 900 degrees Cent. (1650 degrees Fahr.) for 25 minutes and then cooled in still air. If it be assumed that the difference in the grain size of the pearlite in these normalized samples reflects the difference which existed in the size of the austenite grains in these steels at 900 degrees Cent. (1650 degrees Fahr.), then it can be said that at 900 degrees Cent. (1650 degrees Fahr.) the size of the austenite grains in the "fine-grained" Steel A (Fig. 3) was less than that of the austenite grains in the "coarse-grained" Steel B (Fig. 4). Other things being equal, the smaller the actual grain size of metals and alloys, the greater their resistance to deformation. It is not surprising, therefore, that at 900 degrees Cent. (1650 degrees Fahr.) the "fine-grained" steel should resist forging more than the "coarse-grained" steel.

Figs. 5 and 6 show the structures of samples which had been heated to 1025 degrees Cent. (1877 degrees Fahr.) for 25 minutes

and then cooled in still air. From these photomicrographs it might be inferred that the size of the austenite grains at 1025 degrees Cent. (1880 degrees Fahr.) in both steels was about the same. It would be expected, therefore, that at this temperature the two steels would be of equal forgeability. However, the "fine-grained" was rather more forgeable than the "coarse-grained" steel.

The forgeability of the "fine-grained" steel increased over that

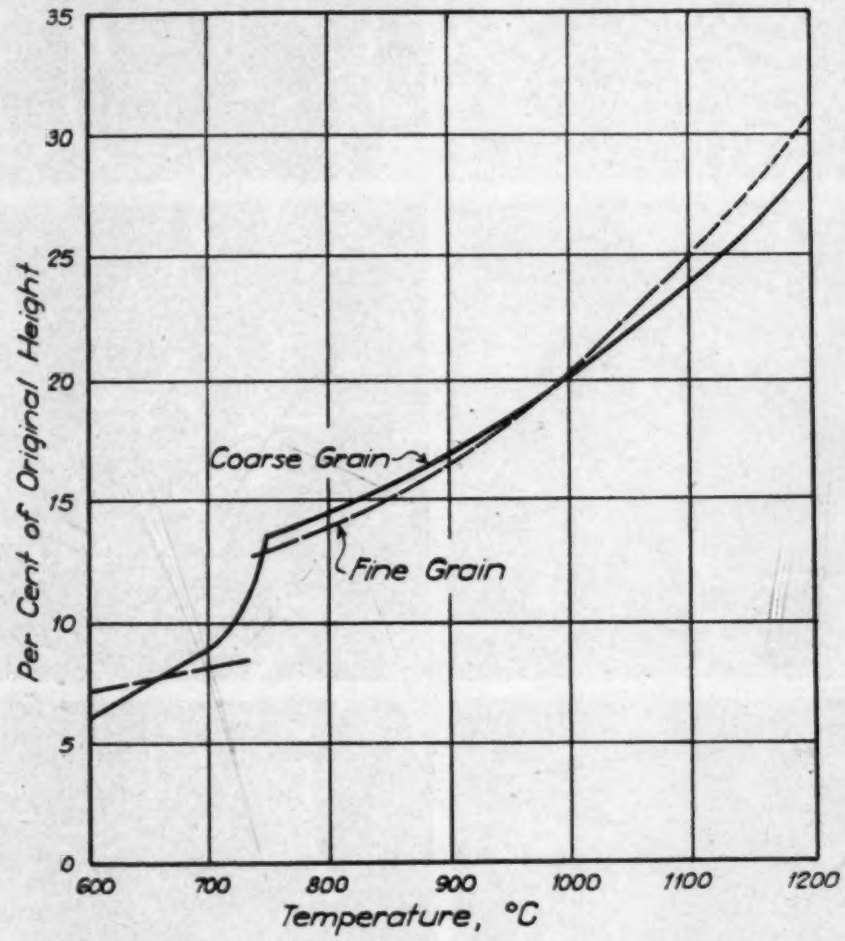

Fig. 2—Forgeability-Temperature Curves for Coarse-Grained and Fine-Grained Steels of Approximately the Same Carbon Content.

of the "coarse-grained" steel as the forging temperature was raised above 1025 degrees Cent. (1880 degrees Fahr.). It was not surprising to find, therefore, that the grain size of the pearlite in the inherently "fine-grained" steel, normalized at 1150 degrees Cent. (2100 degrees Fahr.), was larger than that in the inherently "coarse-grained" steel, normalized at the same temperature. This difference in grain size is brought out in Figs. 7 and 8, the former showing the

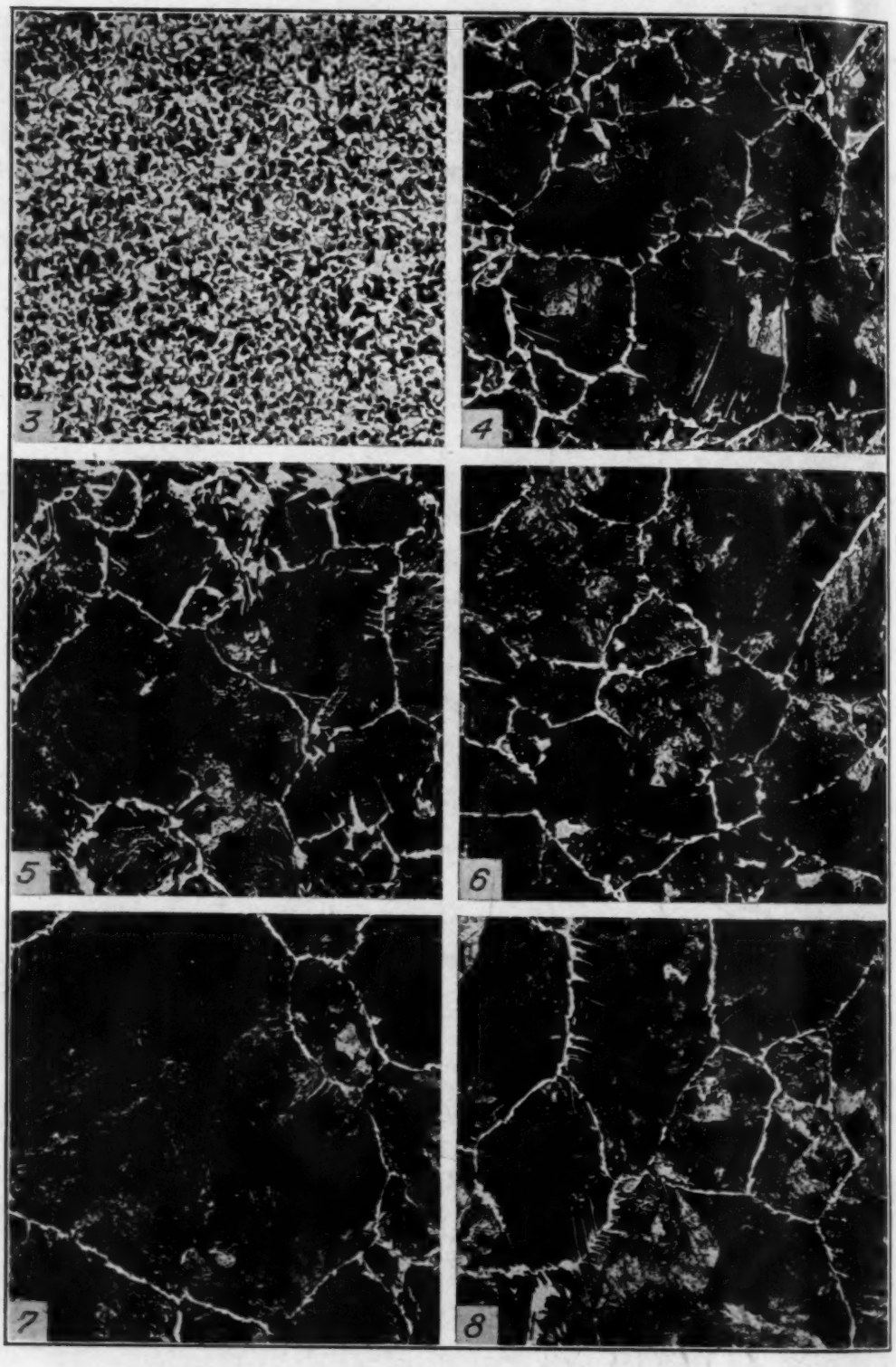

Fig. 3—Fine-Grained Carbon Steel, Normalized at 900 Degrees Cent. × 100.
Fig. 4—Coarse-Grained Carbon Steel, Normalized at 900 Degrees Cent. × 100.
Fig. 5—Fine-Grained Carbon Steel, Normalized at 1025 Degrees Cent. × 100.
Fig. 6—Coarse-Grained Carbon Steel, Normalized at 1025 Degrees Cent. × 100.
Fig. 7—Fine-Grained Carbon Steel, Normalized at 1150 Degrees Cent. × 100.
Fig. 8—Coarse-Grained Carbon Steel, Normalized at 1150 Degrees Cent. × 100.

structure of the "fine-grained" steel (now relatively coarse), the latter that of the "coarse-grained" steel (now relatively fine).

These experiments, it is believed, serve to emphasize how important are the effects of austenite grain size, in contradistinction to inherent grain size, upon the forgeability of carbon steels. One is led to suggest that any elements or compounds (whether in solid solution or not) which reduce the austenite grain size of steel at any temperature will decrease its forgeability at that temperature. In this connection, further reference to Fig. 1 of this paper may be of interest. From this it might be inferred that, while vanadium has the effect of lowering the grain size of steel at low temperatures, and of thus decreasing its forgeability, this effect is lost when the temperature of the steel exceeds about 1000 degrees Cent. (1830 degrees Fahr.).

Cor-Ten versus Straight Carbon Steel

Standard samples of Cor-Ten and of carbon steel (1-inch diameter by 1-inch high) were cut from bars of the analyses and mechanical properties shown below. The results of the forgeability tests on these two steels, together with those on another carbon steel (steel P), referred to in the author's previous paper, and of which the analysis also appears below, are shown in Fig. 9.

	Cor-Ten	Carbon Steel	Steel P
Carbon	0.10 per cent	0.095 per cent	0.075 per cent**
Manganese	0.30 per cent	0.38 per cent	0.44 per cent
Sulphur	0.029 per cent	0.031 per cent	0.037 per cent
Phosphorus	0.151 per cent	0.016 per cent	0.018 per cent
Silicon	0.802 per cent	0.077 per cent	0.003 per cent
Copper	0.46 per cent	....	....
Chromium	1.05 per cent	....	....

	Cor-Ten	Carbon Steel	Steel P
Limit of proportionality (pounds per square inch)	25,000*	20,000	
Yield point (pounds per square inch)	62,250*	45,000	Not
Tensile strength (pounds per square inch)	80,000*	54,100	
Elongation (per cent on 2 inches)	33½*	39	tested
Reduction of area (per cent)	63¼*	73½	

(*Mean of two tests.)

(**Repeat analyses for carbon were made at the Ontario Research Foundation on Oct. 28, 1935.)

The curves in Fig. 9 indicate clearly the increased resistance to deformation at all temperatures up to 1200 degrees Cent. (2190 de-

grees Fahr.), which is conferred upon low carbon steel by the simultaneous presence of chromium, copper, phosphorus, and silicon. At temperatures above 875 degrees Cent. (1605 degrees Fahr.) the forgeability of Cor-Ten, which, it may be noted, is a fine-grained

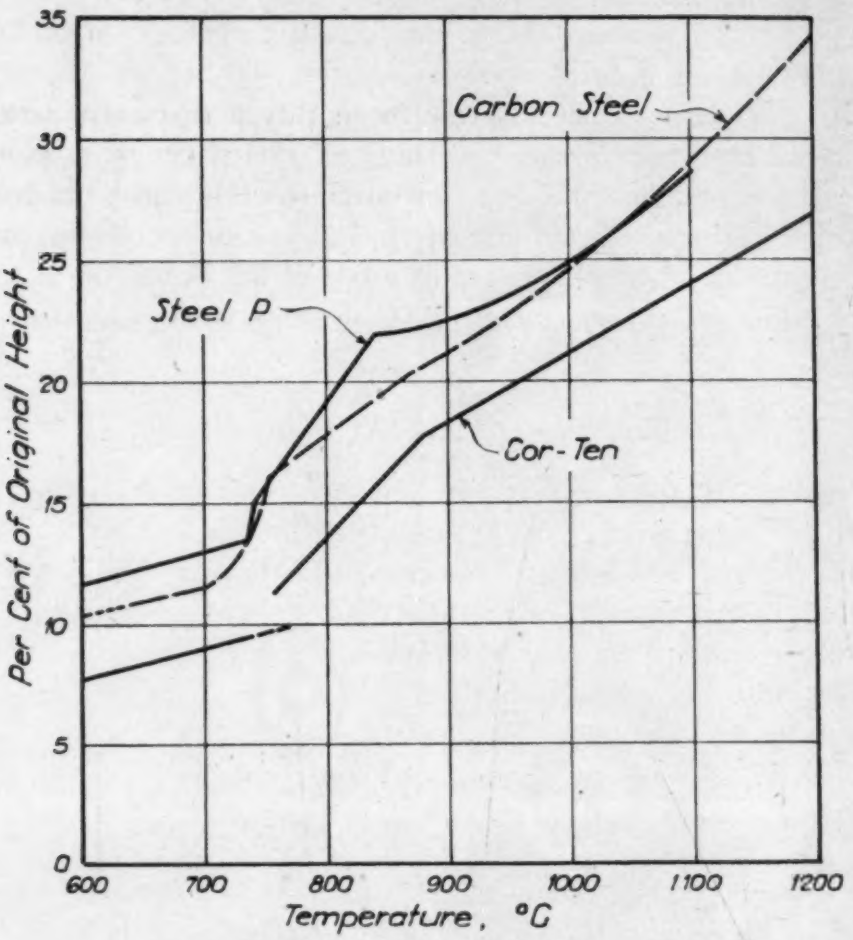

Fig. 9—Forgeability-Temperature Curves for Cor-Ten, a Carbon Steel of Approximately Equal Carbon Content, and Steel P (See Text).

steel, is not much different from that of Man-Ten (see following section), which in turn resembles closely that of a steel containing about 0.40 per cent of carbon (see Fig. 13). Hence, broadly speaking, one may say that Cor-Ten at temperatures above A_3 has a forgeability similar to that of a 0.40 per cent carbon steel.

At temperatures intermediate between about 735 degrees Cent. (1355 degrees Fahr.) and 875 degrees Cent. (1605 degrees Fahr.) the forgeability of Cor-Ten is distinctly less than that of a 0.40 per cent carbon steel, the maximum difference in forgeability between the

two steels occurring at about 750 degrees Cent. (1380 degrees Fahr.) —the A_1 point.

The curve for steel P, abstracted from Fig. 3 of the author's previous paper, is here reproduced to show what wide variations in forgeability are possible in low carbon steels of approximately the same carbon content. It is not impossible that the slightly lower carbon (0.075 per cent) and distinctly lower silicon (0.003 per cent) contents of steel P, as compared with those of the 0.095 per cent

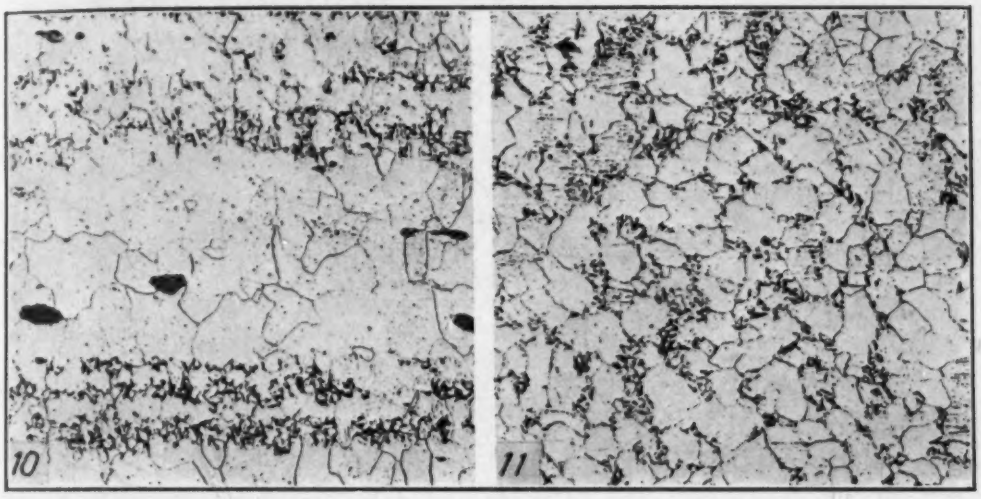

Fig. 10—Photomicrograph of Steel P, for Comparison with Fig. 11; Showing Banding and Relatively Large Grain Size in Ferritic Areas. × 100.

Fig. 11—Photomicrograph of 0.095 Per Cent Carbon Steel, for Comparison with Fig. 10; Showing Uniformity of Structure and Relatively Small Grain Size. × 100.

carbon steel, account for its superior forgeability over the temperature range 755-1025 degrees Cent. (1390-1880 degrees Fahr.). On the other hand, as may be seen by reference to Fig. 10 of this paper, steel P showed banding, the grains in the ferritic areas being distinctly larger than those uniformly distributed throughout the 0.095 per cent carbon steel (Fig. 11), while those elsewhere were about the same size. It is not impossible, of course, that the banding of steel P is related to its lower carbon and silicon contents, and, if so, to the extent that it is related, the superior forgeability of steel P might be said to be due to its composition. But until such a relationship is proved, all one can say is that the superior forgeability of steel P is probably due to its relatively large grain size.

Man-Ten versus Straight Carbon Steel

The results of forgeability tests made on Man-Ten (medium-

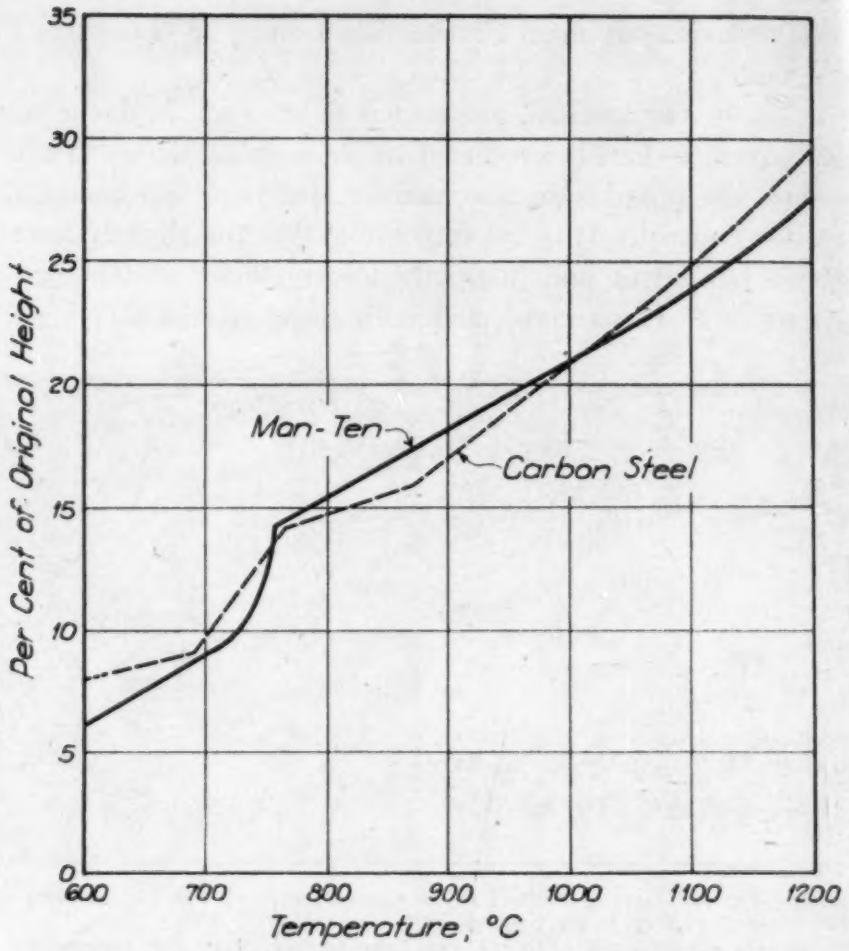

Fig. 12—Forgeability-Temperature Curves for Man-Ten and a Carbon Steel of Approximately Equal Carbon Content.

manganese steel) and on a carbon steel of similar carbon content are shown in Fig. 12. Analyses of these steels are given below.

	Man-Ten	Carbon Steel (for comparison)
Carbon	0.31 per cent	0.30 per cent
Manganese	1.63 per cent	0.73 per cent
Sulphur	0.030 per cent	0.021 per cent
Phosphorus	0.017 per cent	0.020 per cent
Silicon	0.20 per cent	0.18 per cent
Copper	0.01 per cent	....
Limit of proportionality (pounds per square inch)	26,250*	17,500*
Yield point (pounds per square inch)	57,250*	53,500*
Tensile strength (pounds per square inch)	95,200*	79,060*
Elongation (per cent on 2 inches)	29¼*	31½*
Reduction of area (per cent)	64½*	61*

(*Mean of two tests.)

At temperatures between A_1 and about 1005 degrees Cent. (1840 degrees Fahr.) the Man-Ten samples forged more easily than the carbon steel samples, but at above about 1005 degrees Cent. (1840 degrees Fahr.) their relative forgeabilities were reversed. The grain size of the carbon steel, which was reported to be smaller than that of Man-Ten might account for its higher resistance to deformation over the temperature range A_1-1005 degrees Cent. (1840 degrees Fahr.), but could not explain its lowered resistance to deformation

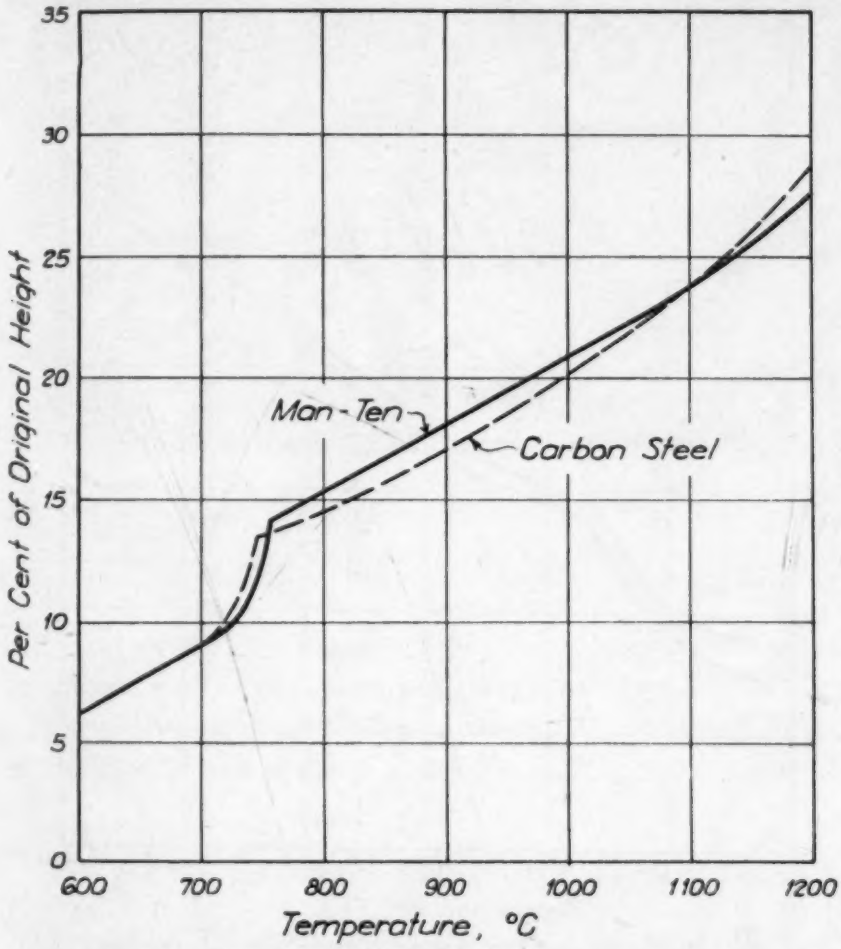

Fig. 13—Forgeability-Temperature Curves for Man-Ten and a Coarse-Grained Carbon Steel of Approximately Equal Tensile Strength at Room Temperature.

over the temperature range 1005-1200 degrees Cent. (1840-2190 degrees Fahr.). Microscopic examination of samples of these steels forged at 875 degrees Cent. (1605 degrees Fahr.) confirmed that the grain size of the carbon steel at this temperature was less than that of the Man-Ten; but examination of samples which had been forged at 1200 degrees Cent. (2190 degrees Fahr.) showed the grain size

of the former at this temperature to be greater than that of the latter. Hence, at some temperature between 875 degrees Cent. (1605 degrees Fahr.) and 1200 degrees Cent. (2190 degrees Fahr.) the grain sizes of these steels must have been the same, and probably this temperature was 1005 degrees Cent. (1840 degrees Fahr.), at which the steels were equally forgeable.

A comparison has also been made between Man-Ten and what was reported[2] to be a coarse-grained carbon steel of a tensile strength

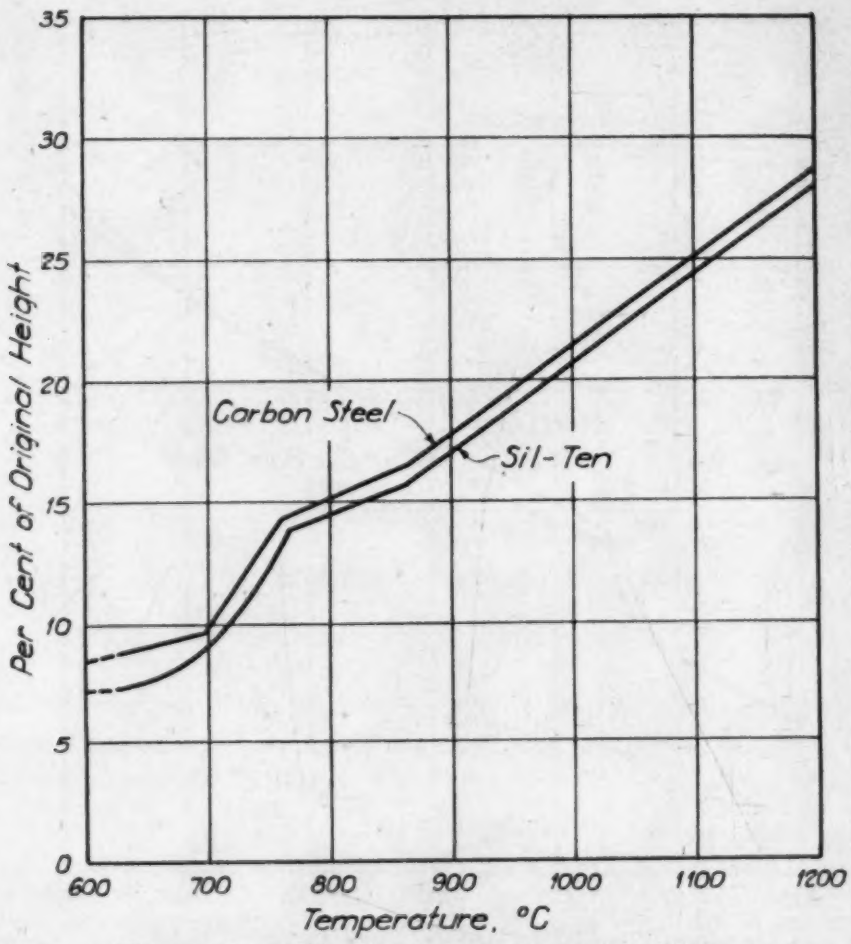

Fig. 14—Forgeability-Temperature Curves for Sil-Ten and a Carbon Steel of Approximately the Same Carbon Content.

equal to that of Man-Ten. This steel had the following analysis and mechanical properties.

Carbon	0.42 per cent
Manganese	0.74 per cent
Sulphur	0.028 per cent
Phosphorus	0.020 per cent
Silicon	0.24 per cent

[2]By Research Department, Carnegie-Illinois Steel Corp.

Limit of proportionality (pounds per square inch)	22,500*
Yield point (pounds per square inch)	50,500*
Tensile strength (pounds per square inch)	90,675*
Elongation (per cent on 2 inches)	28¾*
Reduction of area (per cent)	54½*

(*Mean of two tests.)

The forgeability-temperature curves for these two steels are shown in Fig. 13, whence it will be gathered that, compared with straight

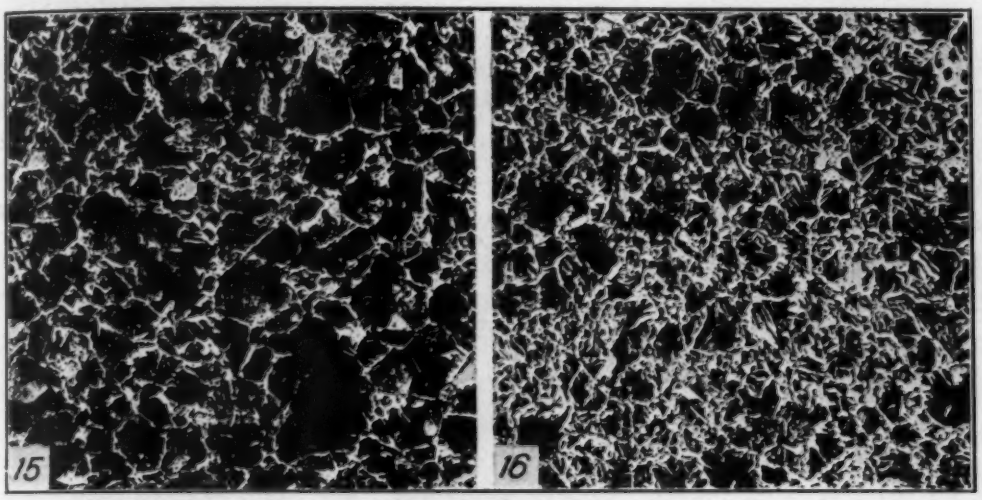

Fig. 15—Photomicrograph of Sil-Ten, Forged at 925 Degrees Cent. (1700 Degrees Fahr.), for Comparison with Fig. 16; Showing Relatively Large Grain Size of Pearlite. × 100.

Fig. 16—Photomicrograph of Carbon Steel, Forged at 925 Degrees Cent. (1700 Degrees Fahr.), for Comparison with Fig. 15; Showing Relatively Small Grain Size of Pearlite. × 100.

carbon steel of about 10 points higher carbon content and somewhat lower (about 6 per cent) tensile strength at room temperature, Man-Ten is slightly softer at all temperatures up to about 1100 degrees Cent. (2010 degrees Fahr.) and slightly harder at higher temperatures.

Sil-Ten versus Straight Carbon Steel

The results of forgeability tests on Sil-Ten (silicon structural steel) and on a carbon steel of approximately the same carbon content are shown in Fig. 14. The analyses and mechanical properties of these two steels are given below:

	Sil-Ten	Carbon Steel (for comparison)
Carbon	0.36 per cent	0.35 per cent
Manganese	0.68 per cent	0.42 per cent
Sulphur	0.024 per cent	0.029 per cent
Phosphorus	0.017 per cent	0.015 per cent
Silicon	0.22 per cent	0.098 per cent

Limit of proportionality (pounds per square inch)	18,750*	15,000*
Yield point (pounds per square inch)	51,050*	43,850*
Tensile strength (pounds per square inch)	87,625*	76,875*
Elongation (per cent on 2 inches)	28*	30¾*
Reduction of area (per cent)	54½*	52*

(*Mean of two tests.)

The curves show Sil-Ten to be uniformly less forgeable at all temperatures between A_1 and 1200 degrees Cent. (2190 degrees Fahr.) than carbon steel of about the same carbon content. The grain size of Sil-Ten at all temperatures was larger than that of the carbon steel, as may be seen, for example, by comparing Figs. 15 and 16, the former showing the structure of a Sil-Ten sample forged at 925 degrees Cent. (1700 degrees Fahr.), the latter that of one of the carbon steel samples forged at the same temperature. From this it may be inferred that the effect of silicon upon the forgeability of steel, while not profound, may be quite appreciable, since it may counterbalance that of its actual grain size, the importance of which has already been emphasized.

Conclusions

1. Grain size has a measurable effect upon the forgeability of steels of low and medium carbon content.

2. The forgeability of Cor-Ten is distinctly less than that of carbon steel of the same carbon content. It can be said, therefore, than the substitution of about 0.75 per cent silicon, about 0.50 per cent of copper, about 1 per cent chromium, and about 0.15 per cent phosphorus for about 2.5 per cent iron in a steel containing approximately 0.1 per cent of carbon reduces its forgeability at all temperatures up to about 1200 degrees Cent. (2190 degrees Fahr.). The fact that Cor-Ten is a fine-grained steel may have an important bearing upon its behavior in this connection.

3. The difference in forgeability between Man-Ten (1.63 per cent manganese) and a carbon steel of approximately the same carbon content (0.73 per cent manganese) is small. In the present tests the grain size of the carbon steel was found to be less than that of Man-Ten at temperatures below about 1005 degrees Cent. (1840 degrees Fahr.), and greater at temperatures above that point. The forgeability of the carbon steel was found to be less than that of Man-Ten

at temperatures below, and greater than that of Man-Ten at temperatures above, 1005 degrees Cent. (1840 degrees Fahr.).

4. Up to about 1100 degrees Cent. (2010 degrees Fahr.), the forgeability of Man-Ten was slightly greater than that of a coarse-grained carbon steel (0.42 per cent carbon) of approximately equal tensile strength at room temperature. At higher temperatures the carbon steel was slightly softer than Man-Ten. However, the differences in forgeability between the two steels were relatively unimportant.

5. At all temperatures between A_1 and 1200 degrees Cent. (2190 degrees Fahr.), Sil-Ten (0.36 per cent carbon; 0.22 per cent silicon; 0.68 per cent manganese) was slightly less forgeable than a carbon steel of about the same carbon content (0.35 per cent carbon; 0.098 per cent silicon; 0.42 per cent manganese). The main cause of the difference in forgeability between the two steels appeared to lie in the difference in actual grain size.

Acknowledgments

The author wishes to express his appreciation of the help given him in this work by E. S. Taylerson, who was primarily responsible for supplying the finished samples of steel for test; to Dr. H. B. Speakman, whose encouragement and constructive criticism have been invaluable; to Dr. G. S. Farnham, to whom are due the photomicrographs which illustrate the paper; and to Gordon Coleman, upon whom fell the burden of most of the experimental work involved in this investigation.

DISCUSSION

Oral Discussion

C. L. Clark:[1] The ductility values obtained from short-time tensile tests at temperatures of 1400 to 2300 degrees Fahr. indicate that certain steels of the type herein considered may possess hot-short characteristics over certain portions of the temperature range. I am wondering if Mr. Ellis' work has confirmed this?

O. W. Ellis: My experiments have not brought out the hot-short characteristics of any of these steels. The percentage reductions in height of normal 1-inch samples are relatively small. Had normal ½-inch samples been used, the percentage reductions in height would have been sufficient, in all probability, to cause cracks to develop in the samples when forged at temperatures within the hot-short ranges. Experiments which we carried out with normal

[1]Department of Engineering Research, University of Michigan, Ann Arbor, Mich.

½-inch samples on electrolytic iron brought out quite clearly the hot-short ranges for this material.

J. F. HARPER:[2] I would like to ask Mr. Ellis if, in the samples for forgeability tests, the pieces were all cold-rolled steel taken from small sizes? I wonder if the influence of the former forging or rolling action has been taken into account. I feel that there is a possibility of error creeping in in determining forgeability of steel, without having the previous history of such work.

Speaking from practical experience, I know the degree of forgeability, as recognized in shops, is much altered by forging which has taken place prior to the one under discussion.

O. W. ELLIS: The samples tested by us were all cut from hot-rolled steel bars of relatively small dimensions. No attempt was made to correlate the results of the tests with the previous history of the materials from which the bars were rolled. We are in full agreement that it might be worth while to determine the forgeability, shall we say, of one and the same steel—first, in ingot form, then in billet form, and finally in rolled form. We are taking steps to carry out experiments along these lines.

J. L. BURNS:[3] Are the results below the critical range attributable to the use of material with a fine as-rolled grain size? It would seem that on the size used (1-inch round) a fine as-rolled grain size might be expected if previous heat treatments were not performed.

O. W. ELLIS: The results of the tests carried out at temperatures below the critical range are closely related to the grain size of the as-rolled material. In previous tests (O. W. Ellis: "Further Experiments on the Forgeability of Steel," TRANSACTIONS, American Society for Metals, Vol. 21, 1935, p. 673) we found that a steel containing 0.68 per cent carbon was much more resistant to deformation at temperatures below A_1 than a steel containing 0.85 per cent carbon, whereas at temperatures above the critical range the steels behaved in what might be called a normal fashion. In our experiments we made no attempt to anneal the bars before machining the test pieces, since we were more interested in the behavior of these steels at temperatures above the critical ranges than at temperatures below them.

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[2]Globe Union, Inc., Milwaukee.

[3]Metallurgical Department, Republic Steel Corp., Chicago.

NOTES ON CONTINUOUS GAS CARBURIZING

By R. J. Cowan

Abstract

The first part of this article is devoted to a discussion of various methods used to ascertain the effect of gas flow upon the rate of carburization and on case depth. These results were obtained from a number of industrial installations so that the results would be applicable to existing commercial operations. One definite result was the fact that when the rate of gas flow is increased to the point where it results in agitation in the muffle, it causes a nonuniform carburization. The use of propane and butane as the hydrocarbon carburizing gas is discussed and covers the various obstacles which are encountered and which may be overcome by a reversal of the usual flow of the carburizing gases.

The latter part of the article summarizes a group of experiments conducted, using, in turn, city gas, cracked gas, and natural gas under various operating conditions, to determine the effect of various atmospheric conditions upon the process of continuous gas carburizing. At the end of the article is listed a series of conclusions which are the results of these tests.

THE method of carburizing by gas in which the work enters at one end and discharges at the other end of an elongated muffle chamber was proposed a number of years ago.[1] It has been in successful commercial use since that time. A vast amount of information regarding the operation of these units has been accumulating. It is believed that some of this information will be of general interest as applying not only to the operation of this process but also to the art in general. The following paper details certain of these observations. Some of these facts seem to be at variance with observations made by others covering different applications. A discussion of these ideas should prove to be valuable to all phases of the art as an attempt to assist in the pursuit of truth and render an ancient art more positive and scientific.

[1] R. J. Cowan, "Development of Continuous Gas Carburizing," *Transactions*, American Institute of Mining and Metallurgical Engineers, Iron and Steel Div., Vol. 95, 1931, p. 386-403.

A paper presented before the Eighteenth Annual Convention of the Society held in Cleveland, October 19 to 23, 1936. The author, R. J. Cowan, is metallurgical engineer, Surface Combustion Corp., Toledo, Ohio. Manuscript received June 15, 1936.

Effect of Gas Flow

A great deal has been written about the effect of gas flow upon the rate of carburization. This is known to affect both the carbon concentration and depth of case. A rapid movement of gas over a metal surface causes a rapid carburization of the metal with the formation of a higher carbon content in the surface layers of the case as the flow increases. One of the best references is the work of Bramley and Jenkings.[2] The results obtained by varying the flow of carbon monoxide gas has been summarized by these investigators in the form of a chart which is presented herewith after having been replotted to a different scale. An examination of this chart indicates

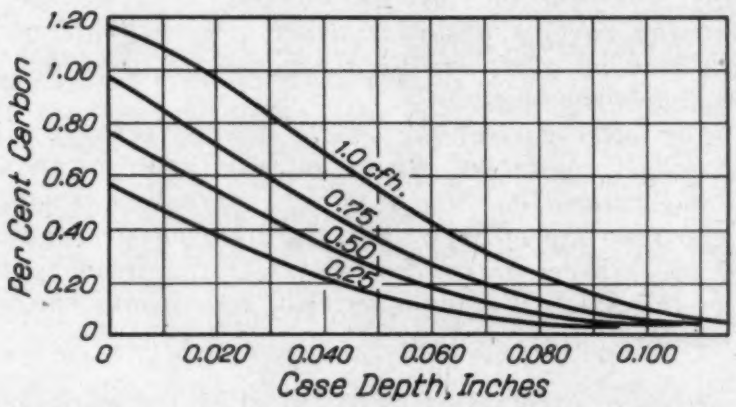


Fig. 1—Bramley and Jenkings Data. CO Carburizing, 20 Hours at 1742 Degrees Fahr.

clearly that the carbon concentration in the case varies in accordance with the gas flow and that the depth of case varies also with the gas flow. See Fig. 1.

In our own laboratory there has been carried through a rather elaborate set of tests, using ten different steels of the common carburizing variety to determine the effect of different gas flows upon the case depth when using propane as the carburizing gas. It was found that there was both a minimum and maximum flow beyond which we did not obtain good carburization. This work was done in a quartz tube 1 inch in diameter within an electric laboratory furnace of the usual type using two pieces of each steel for the test specimens. Under these conditions it was found that a flow of 0.24 cubic feet per hour was too slow for best results; that 1.85 cubic feet per hour was too fast for most steel; and that the best rate was about 0.8 cubic

[2]Bramley and Jenkings, "The Gaseous Cementation of Iron and Steel," *Carnegie Scholarship Memoirs*, Iron and Steel Institute, Vol. 15, 1926, p. 39.

feet of gas per hour. This was true for all steels except the 3.5 per cent nickel-molybdenum (S. A. E. 4815) in which a flow of 2.0 cubic feet per hour was found to be more satisfactory. In this work no attempt was made to determine carbon concentration, but only to note the effect on case depth.

In view of these results and of others along similar lines that are well known, it has frequently been proposed to use this idea in the continuous process by increasing the flow and causing an agitation of the gases within the muffle. This has been done in different ways in a number of industrial installations so that the results obtained are such as would be applicable to existing commercial operations.

In one instance in which the carburizing gas entered wholly at the charge end of the muffle, the total gas flow was increased from 150 cubic feet per hour to 300 cubic feet per hour. Under these conditions it was found that the carburized case at the top of the load was much deeper than at the bottom of the load, that is, the deepest case obtained was about 0.005 inch greater than the previous average with the lower flow, but at the bottom of the load the case had dropped from about 0.060 to about 0.035 inch. It is readily seen what has taken place in this experiment; the increased gas flow has channeled along the top of the muffle and has prevented the circulation of gas within the mass of the load of work, thereby producing very nonuniform results.

In another instance where the muffle was provided with a number of gas inlets, at the top, the total gas flow was increased from 160 cubic feet per hour to 250 cubic feet per hour. This gas was distributed uniformly through each of the inlets. The results in this case were decidedly variable and nonuniform. The shallow cases and the deep ones were not found in any definite location on the tray. The deepest case obtained was only slightly greater that the normal case with the lower gas flow.

In another instance the gas inlets, which are usually positioned in the center of the muffle at the top, were offset in such a way as to produce an agitation of the gas currents within the muffle at each point of entry. The results obtained by this method were also very nonuniform, with many instances of shallow cases within the mass of the load.

In another instance where the work was being carburized in an intermittent type furnace in which the gas is made to enter the muffle at a number of points in opposite sides, an increase of the gas flow

brought about a concentration of the higher case depth within the upper part of the load and much shallower cases in the lower part next to the tray.

These tests reveal the fact that an increased gas flow has resulted, in each case, in the production of very nonuniform carburization. This is due to the fact, perhaps, that a rapid movement of the carburizing gases over parts of irregular shape produces on some portions a deep case and on other portions a shallow case in accordance with the movement of the gases over the surfaces exposed. An attempt to increase the movement of these gases seems to result in stratification which would be very difficult to overcome even by means of extreme agitation. Any attempt, therefore, to increase the rate of carburization by a more rapid circulation of gases within the muffle seems to be subject to severe limitation from the standpoint of uniform case results based on uniform gas movement.

To obtain uniform carburization which will extend to all parts of the load within the cross section of the muffle, it has been found to be desirable to cause the gas to enter the muffle in such a way as to avoid agitation. The gas circulation that occurs within the muffle is due, therefore, to the action of convection currents which bring about by means of their uniform movement a positive circulation that is in accordance with the temperature distribution within the muffle and which completely surrounds all surfaces of the parts that are being carburized and produces uniform results.

Propane, Butane and Coke Formation

The use of propane or butane as the hydrocarbon carburizing gas is being carried on in a number of installations. A number of interesting obstacles have been encountered and overcome in making these applications which seem to be inherent in the properties of these gases themselves. The studies that have been carried on and the various means that have been adopted to overcome the difficulties encountered make up a long chapter in the development of this process. The obstacles have been in connection with the formation of tar which occurs when these gases are heated. The slow heating that takes place in that part of the furnace where the work is coming to temperature, brings about a polymerization of the hydrocarbons and the formation of liquid condensable products which tend to accumulate in any convenient pocket that may be found in the steel itself. These tars so deposited are then carried into the high temperature carburiz-

ing zone along with the work where they result in the formation of hard carbon or coke upon the surface to be carburized, thereby interfering with the uniform carburization of the metal surface. This coke is metallic in luster and seems very similar to iron scale in appearance so that it is easily mistaken for iron scale. It may usually be removed from the surface while still retaining the shape of the piece.

It frequently happens that no coke is apparent on the carburized work as it leaves the furnace although there has been very spotty carburization. A close examination of the surface of the piece, however, will frequently reveal markings caused by coke which has disappeared before the part leaves the furnace. In every single piece where a normal case has dropped off suddenly to a shallow one followed again by a normal case depth, the cause has been found to be associated with coke formation in the successive stages of the operation.

This condition is found to be aggravated in the use of propane and butane of those commercial grades that contain liberal percentages of propylene or butylene. In these cases the formation of coke is excessive and results in very spotty carburization. The tar so formed collects in the heating zone of the furnace and causes an objectionable operating condition. By using such grades of these gases as are free from the corresponding unsaturated hydrocarbons, this condition is relieved but not eliminated, since these objectionable tars will be formed also, but in lesser amount, by the slow heating of the pure gases.

Through the courtesy of a producing company, we were given a supply of propane of two varieties, one being a gas containing not more than 2 per cent propylene, and the other containing 35 per cent propylene with the balance propane. A study in our laboratory, based on the use of these two gases, showed that it was possible to use either of them for the formation of cracked gas but that when added directly to the muffle along with cracked gas to accelerate carburizing, the gas containing the larger percentage of the unsaturated hydrocarbon caused an active coke formation even when diluted with cracked gas in rather large proportions. This experience has been confirmed by a number of observations on full size industrial units. In each case the gas containing the unsaturated hydrocarbons produced nonuniform carburizing as a result of coke formation

The exact nature of these liquid condensable products, which

separate as tars and form coke at higher temperatures, has not been investigated in detail; a principal concern has been with working out means for overcoming the objectionable characteristics.

A combination of two separate ways has been used to overcome coke formation when using the higher hydrocarbon gases. One is by dilution with a carburizing gas high in hydrogen and the other is by introducing most of this gas into the high temperature zone of the muffle. The high hydrogen diluent lowers the tendency toward tar formation by retarding polymerization and bringing about at high temperatures pyrolysis of the hydrocarbon into hydrogen and carbon that is free from the objectionable features. By introducing most of the gas into the high temperature region, the thermal breakdown is so rapid that the unsaturated hydrocarbons are unable to persist and the lower and more stable saturated series bring about carburization without these interfering reactions.

A change in mechanical construction of the unit has been necessary to make it possible to take advantage of these facts. Instead of forcing all of the carburizing gas into the muffle at the charging end, a number of additional inlets have been placed at regular intervals along the top of the muffle from one end to the other of the furnace. These may be used together or singly. Provision has been made to vary the proportions of the gases which enter the different inlets. These proportions affect both the total volume of gases used and the carburizing concentration of these gases at each point of entry. This change has made it possible to accomplish several objectives at the same time. In the first place, it reduces the volume of gases admitted at any single point, thus avoiding the high velocity movement of these gases and allowing normal convection currents to be more effective in producing uniform carburization. It makes it possible also to control the carbon in the carburized case by proportioning the gas concentration at each inlet in accordance with the carbon concentration desired. By this means there may be used in the heating zone a very great dilution of propane or butane which will prevent tar from forming in the early stages of the heating operation.

It has been pointed out that a reversal of the usual flow of the carburizing gases would overcome the obstacles encountered in the use of propane and butane for carburizing. As originally planned, the gases were made to enter at the charge end of the muffle and leave at the discharge end. By reversing this flow the higher hydrocarbon gases would be entering at the high temperature zone in such

a way as to avoid tar formation and in their progressive movement toward the charge end, where they would leave the muffle, would be broken down completely or to the more stable hydrocarbon form which would not deposit tar on the work in the heating zone. When this idea is coupled up with the use of additional gas inlets extending along the top of the muffle, it would seem to have merit although it has not been adopted in any commercial installation. When used for a time in conjunction with city gas, the large volume of gas required caused distortion of the carburized parts from the chilling action of the cold gases impinging upon these parts and nonuniform carburization resulting from high velocity gas travel.

The use of carburizing gases in reverse flow, so that they are moving in a direction countercurrent to that of the part being carburized, does not alter the original conception of three reaction zones within the muffle of this furnace. Although the gases are flowing in the opposite direction, it is still found that the first zone or heating zone of the furnace is characterized by the deposition of carbon upon the work to be carburized. Since in this case low temperatures are involved, this carbon will come from the presence of carbon monoxide as well as hydrocarbon in the gases. The separation of this carbon will result in the formation of carbon dioxide which will react in the second zone at high temperatures with the carbon deposited in the first zone, to produce carbon monoxide at the metal surface. The third zone of the furnace was originally referred to as the diffusion zone and this will still be effective since diffusion will take place toward the core of the metal being treated while at the same time a high carbon hypereutectoidal case will be packed into the surface of the material. It is probable that for the carburization of abnormal steels, the reverse flow of carburizing gases would be more effective than the usual procedure, for by this means the rich carburizing hydrocarbon gases would be brought into contact with the material immediately before being discharged so as to avoid the presence of oxygen containing gases.

Experimental

In order to determine the effect of various atmospheric conditions upon the process of continuous gas carburizing there was set up in the laboratory a small furnace adapted for continuous operation in which material could be carburized in a controlled atmosphere and allowed to cool slowly before removing into the air. The parts

to be carburized were placed in steel trays, which were charged at one end of the muffle, moved progressively along the furnace length and discharged at the opposite end of the muffle. The details of the furnace used are shown in Fig. 2.

This furnace was operated continuously for a considerable period of time for the carburization of samples of steel which were then polished and examined by the microscope to study the depth and

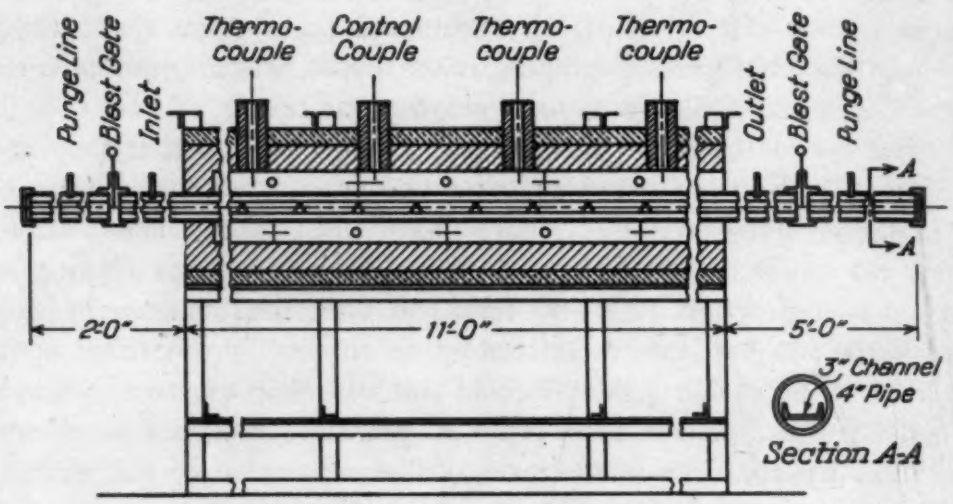


Fig. 2—Set-Up for Carburizing Test.

character of the carburized case obtained under the different conditions. For these studies there were used samples of steel S. A. E.-1020-90 Mn. The time at temperature was adjusted so as to produce on these specimens a rather deep case of approximately 0.070 inch. This was done in order to avoid conclusions based on shallow case depths. There were 20 trays in the furnace at one time each with a test specimen; therefore, when it was decided to make a change of any kind there was a period of time when certain of the samples within the furnace were being subjected to both treatments. In the accompanying graph (Fig. 3) an attempt is made to show by means of shaded areas and the cross hatch lines just where these transition periods occur between tests.

The following series of tests were run:

1—City gas only, concurrent flow 11 C. F. H.
2—City gas only, concurrent flow 40 C. F. H.
3—City gas only, counterflow 11 C. F. H.
4—Natural gas with flue gas.
5—Cracked gas with propane dehydrated by activated alumina.
6—Same as No. 5 without dehydration.
7—Cracked gas with propane, dew point 60 degrees Fahr.

8—Cracked gas with propane, dew point 75 degrees Fahr.
9—Cracked gas with propane, alloy trays.
10—Cracked gas with propane, alloy muffle.
11—Cracked gas with propane, oxidized trays.

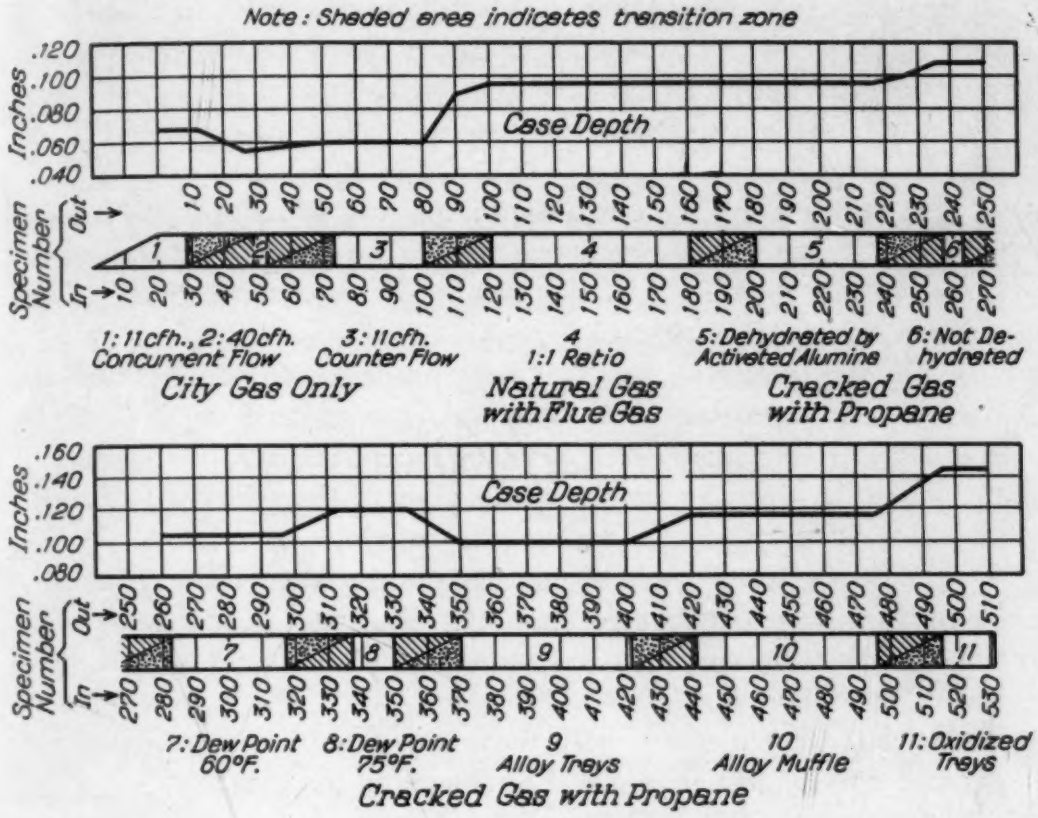


Fig. 3—Series of Carburizing Tests.

At the base of this chart there is designated by the appropriate numbers the different series of tests given above. The transition zones between tests are marked out as already described. The numbers shown represent individual specimens each of which was taken from a single tray. The carburized case depth of each is recorded by a graph placed above each specimen number. The temperature was held at 1750 degrees Fahr. (955 degrees Cent.).

The first three tests of this series represent results obtained by the use of Toledo city gas of the following composition:

	Per Cent
CO_2	2.8
O_2	0.3
CO	8.4
H_2	48.1
Unsaturates	4.1
Methane	33.1
N_2	3.3

These tests show the effect of industrial gas of this composition when used in different ways. Series No. 1 shows the result of concurrent flow in producing a certain depth case. When the velocity of this gas was increased there was a drop in case depth of about 0.010 inch as shown by Series No. 2. In Series No. 3 the original flow was re-established but its direction was changed so that it flowed counter to the work. This also produced a more shallow case than in Series No. 1.

In Series No. 4 there was used a mixture of natural gas and flue gas. As shown by the chart, this resulted in a very considerable increase in case depth, for by this means, the case was increased from about 0.065 to 0.095 inch.

For the remaining series of tests propane was used in conjunction with cracked gas. The flow was standardized at 15 C. F. H. and all conditions maintained constant except the moisture content of the gas so as to make it possible to observe the effects of different moisture content upon the carburizing reactions.

In Series No. 5 considerable pains were taken to dehydrate the gases by means of activated alumina. The dew point of the gases dried by the apparatus used was found by determination to be equivalent to saturation at negative 32 degrees Fahr. The results obtained in this way were identical with those obtained by the use of natural gas and flue gas in Series No. 4 as shown by the graph.

Series No. 6 was conducted the same as No. 5 except that the gases passing through the dehydrating unit were bypassed around the unit without entailing any other change. Series No. 7 was essentially the same except that the dew point was maintained at 60 degrees Fahr. In Series No. 8 the dew point was maintained at 75 degrees Fahr. It is apparent in these series that an attempt was being made to determine the effects of water vapor upon the gas reactions taking place during the process of continuous gas carburizing. The results were different from what had been expected, since it was found that with a progressive increase in moisture content there was also an increase in case depth from 0.096 inch in the dehydrated gas to 0.120 inch in the gas with a dew point of 75 degrees Fahr.

The next two series of tests were carried through for the purpose of determining the effect of heat resisting alloy upon the carburizing reactions.

In Series No. 9 the samples were carburized along with pieces of alloy containing 35 per cent nickel and 15 per cent chromium.

The effect of this alloy in retarding the carburizing rate is clearly indicated by the fact that the case depth dropped from 0.120 to 0.100 inch in the presence of the alloy. The use of alloy in the muffle itself, however, did not show a result as nearly pronounced (Series No. 10) since in this case the depth was increased to 0.116 inch which is almost the same as the result shown in Series No. 8 which is otherwise comparable.

In Series No. 11 the trays were oxidized previous to use. The effect of this operation was apparent at once in that the case depth increased very considerably to a maximum of 0.145 inch which represented the highest rate attained in any of these tests.

A number of unexpected results were found in this program of tests. We were surprised in the first place to find that a higher flow of city gas caused a lowering of the case depth. We were surprised also when the completely dehydrated gas was not found to be an active carburizing gas and when the addition of moisture to this gas raised its carburizing efficiency. The effect of alloy on the carburizing reaction was well known and expected, but it was believed that the use of alloy in the muffle would have about the same effect as alloy in the tray. This proved to be wrong for the effect of alloy in the muffle is rather slight. The use of oxidized trays or of material covered with mill scale is known to have an effect upon the carburizing rate. We did not believe the effect was as great as that indicated.

The conclusions that may be drawn from this series of tests seem to be —

1. The matter of gas flow is important and should be carefully established for each installation.
2. The carburizing gases do not need to be thoroughly dried, but will function efficiently in the presence of considerable water vapor.
3. Alloy trays containing 35 per cent nickel and 15 per cent chromium have an effect upon the carburizing reaction which must be taken into consideration. (Experience has shown that after having passed through the furnace several times and having been thoroughly carburized this alloy effect is greatly reduced.
4. The alloy that is used in the muffle has but very slight effect, if any, upon the carburized material.

5. The presence of mill scale upon work to be carburized accelerates the carburizing rate. This observation has been confirmed in practice.

This paper represents, in fact, the contributions of a number of people. Among the members of the laboratory staff should be mentioned especially the work of F. D. Widner who carried on most of the experimental work. Grateful acknowledgment is made of the suggestions and recommendations of plant metallurgists in the plants where these units have been installed. This work has been under the supervision of E. G. deCoriolis, Director of Research, Surface Combustion Corporation.

DISCUSSION

Written Discussion: By H. L. Daasch, Iowa State College, Ames, Iowa.

The development of continuous carburization methods by Mr. Cowan and his associates is outstanding. The presentation of facts "at variance with observations made by others" for purposes of discussion "as an attempt to assist in the pursuit of truth" is an admirable attitude, yet quite in accordance with past practices of Mr. Cowan.

A study and discussion of the effect of gas flow rates does not seem truly logical or fundamental. The carburization of steel is possible because of the lack of chemical equilibrium. Equilibrium once attained precludes carburization. Whether that equilibrium is attained by virtue of reaction at the steel surface, whether penetration of carbon is by a process of diffusion, or whether the chemical equilibrium is also established by reaction below the surface by diffused gases seems beside the point here. The fact may be accepted that carburization proceeds only while a "carburization potential" (due to lack of equilibrium) exists.

Now, time is necessary to attain such equilibrium. To the writer the important variable because of varied gas flow rate is a varied effective time toward the attainment of equilibrium.

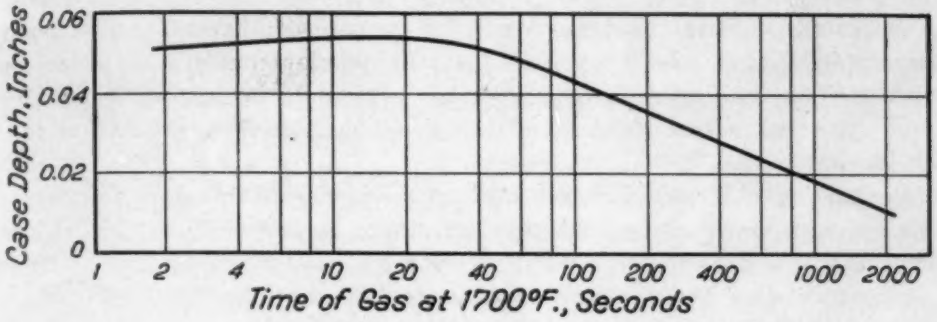
Further, the "carburization potential" may be expected to decrease in an isolated carburizing system as the effective time is increased because of (a) progress of the reaction toward equilibrium and the application of facts stated by the "mass" law and (b) by the effect of heat through the breakdown of many carburizing gases to constituents of lower carburizing power.

The writer therefore feels that flow rates given by Mr. Cowan are important, but would be of more fundamental value if quoted in terms of the time a given volume of gas remained at carburizing temperature.

This thought has been the basis of certain experimental work carried on by the writer and which will be more formally presented at an early date. Briefly, it has been found, for example, that the depth of case produced was a maximum when the carburizing gas had been at heat for 20 seconds. This figure applies to a carburetted blue water gas used in a plain carbon steel

retort at a carburizing temperature of 1700 degrees Fahr. Fig. 1 is offered as typical of results secured. It will be noted that short periods of the gas at heat show less change in carburizing activity than do long periods. It is apparent that the gas heating period can be so shortened as to not permit attainment of carburizing temperature. What the effect of this item may be, with reference to results indicated in Fig. 1, is not known at this time.

It would be interesting to have more complete data from Mr. Cowan relative to the temperature used and the heated length of the quartz tube up



Case Depth—Time of Gas at 1700 Degrees Fahr.
S.A.E. Steel 1020. City Gas Carburizing Medium, 5 Hours at 1700 Degrees Fahr.

to the point where the carburized specimens were located. Gas flow rates quoted on page 844 could then be converted to "Time of Gas at Carburizing Temperature" basis for direct comparison to the above quoted 20 seconds.

The variations in certain commercial units quoted on pages 845 and 846 may be due, as the author states, to "stratification." However, on the basis of the above thoughts, stratification results in varied time of "gas at heat" in the various strata.

The writer has observed the formation of coke and lustrous scale formations when highly carburetted city gases are used as carburizers in stationary retorts. Results of carburization in several hundreds of heats has shown no correlation of the scale with variations in cases produced.

The writer is not clear in regard to statements on page 849 of the paper and should like to inquire how carbon dioxide produced as a result of carbon deposition may enter into the carburizing process. For reversed flow, the CO_2 produced in the heating zone must leave the furnace at the adjacent charging end.

Finally, there may be questions regarding the use of the experimental equipment shown in Fig. 2. Inasmuch as the specimens were cooled in the furnace, the final results are dependent on a period of high temperature carburization plus a period of carburization and diffusion during the cooling cycle. The effect of diffusion would be dependent upon the carbon content of the case which in turn might be a function of the carburizing gases. Further, it would be of interest if data regarding the speed of the specimens as well as the heated and cooling lengths of the retort were known. This would make for a better study of the various parts of the cycle, would develop facts pertaining to the relationship of depth of cases to carburizing time, and would establish the limits of the time of gas at carburizing temperature for various

gas flow rates. These items may not have been within the intended scope of the paper and yet they suggest the far-reaching character of the large amount of work performed by Mr. Cowan.

Written Discussion: By Gordon T. Williams, metallurgist, Cleveland Tractor Co., Cleveland.

Mr. Cowan's paper is a valuable contribution of further data on gas carburizing. We are undoubtedly approaching an understanding of the chemistry and mechanics of the process, and some day all the observations reported from Giolitti to the present will fall into a clear pattern.

A certain definite trend toward increased case depth from left to right is apparent in Fig. 4. Is it possible that the tubular muffle was incompletely conditioned at the beginning of the tests? It would be helpful if the author would state the actual time at heat for comparison with production results with various media.

Guthrie in 1927 and 1928[1] reported on the beneficial effect of a preliminary oxidation with water vapor, and found results to be best with city gas if water-saturated gas were used for 3 minutes, then dry gas for the balance of the run. Is it possible that Mr. Cowan's results are the same, his being a continuous process, and the water perhaps being removed by action on the first pieces it strikes? The reports of Guthrie covered city gas; perhaps the same thing would be true with a more vigorous medium. We found some years ago that 30 per cent air could be admixed with natural gas without decreasing rate but with a reduction in soot. Probably these effects are related.

The author's remark on acceleration due to presence of mill scale on work led us to make a few crude experiments. First, pieces of X-1020 were scaled at 1500 degrees Fahr. and at 1800 degrees Fahr. Thin oxide coatings were produced. One end of each piece was then ground to bright metal. One piece of each temperature of scaling was then carburized in natural gas—flue gas mixture, and one of each in compound. No difference in case depth was found between bright and scaled ends on any of the pieces. Second, a bar of X-1020 forged and cleaned by tumbling was machined over part of its surface, then carburized in the gas mixture. On completion of the cycle, it was observed that there was a heavy soot deposit on the rough surface, only a slight film on the machined portion, but case depth and characteristics were identical on the two sections. Third, two ½ inch holes, 1½ inch apart, were drilled half way through a piece of 1¼ inch S.A.E. 1020; one of the holes was filled with steel scale, the other left open. Carburizing with natural gas—flue gas mixture gave results as before; case uniform in both holes and over entire surface, soot deposit greatest on and near scale.

Perhaps where active gas supply is limited, oxide will by soot deposition accelerate carburizing, but these tests, admittedly crude, seem to discount the beneficial effect in normal gas carburizing.

We will continue to look forward to progress reports from Mr. Cowan on this interesting and practical study.

Written Discussion: By J. F. Wyzalek, Hyatt Bearings Div., General Motors Corp.

The author is to be congratulated upon the practical treatment in his paper

[1] TRANSACTIONS, American Society for Steel Treating, Vol. XII, p. 853; Vol. XV, p. 96.

of a highly technical subject. In general, results recorded and the conclusions drawn parallel our own experience with continuous gas carburizing.

We agree with the author's statement that there is both a minimum and a maximum flow of carburizing gas, beyond which good carburization is not obtained. Our own experience very definitely bears this out. We have found that too low a volume or too slow a movement of the carburizing gas results in shallow depth of case and nonuniform character of case, tending toward low carbon concentration. On the other hand, increasing the flow beyond a satisfactory maximum results, apparently, in pronounced turbulence of carburizing gas in the muffle which seems to create a very pronounced nonuniformity, both in depth and character of case, at the same time resulting in a pronounced carbon deposit on the product with a tendency towards coking. The latter results in soft spots on the quenched work.

To effect best results we have found it necessary to apply the carburizing gas to the muffle at four locations—one at the bottom of the muffle at the charging end, the other three inlets located at the top of the muffle, spaced so as to allow one for approximately each third of the muffle length. It was also found particularly advantageous to have the top inlet tubes specially constructed so as to direct the gas in fan-shaped manner toward the feed end of the furnace, which is reverse of its ultimate flow to the effluent gas opening located on the discharge end of the furnace.

We also found that a greater amount of carburizing gas in a cold zone creates coking, while insufficient amount in the hot zone would tend to create low carbon content in the case or decarburization.

Regarding the relative merits of butane and propane as raw gas additions to cracked gas base, our experience definitely indicates the superiority of natural gas propane. It was found that the use of natural gas butane as a raw gas addition necessitated higher carburizing temperatures to obtain reasonably good results, temperatures below 1750 degrees Fahr. being unsatisfactory, while on the other hand, with raw gas additions of natural propane, temperatures 50 degrees Fahr. lower are found to be satisfactory. This is particularly important in connection with direct quenched product where the amount of retained austenite must be kept at a minimum.

Although the author has not referred to the surface condition of the product to be carburized, we believe a few words on this subject would not be out of order. Our own experience has definitely indicated that a thorough cleaning of the product is absolutely essential toward satisfactory quality of carburized case. We have found that ordinary methods of cleaning are not suitable and the only process found satisfactory was the vapor cleaning method. The presence of grease or oil, and particularly those containing sulphur, we have found to be very detrimental, creating soft spots at which the case is not only shallow but low in carbon. Further, we have found rust of any nature on the work to be very disturbing, immediately upsetting the balance in the furnace and creating nonuniformity in case.

In conclusion it might be well to mention that we, in spite of the points made as being essential to watch closely, have found the process to be very practical and unquestionably very reliable—producing day after day a very uniform depth and character of case in the product being carburized.

Written Discussion: By H. W. McQuaid, metallurgist, Republic Steel Corp., Massillon, Ohio.

I would like to add to this excellent paper of Mr. Cowan's a few observations which might be of interest to other readers. I am certain, from my experience, that the rate of flow is an important factor in obtaining the maximum rate of carburizing, whether in gas or in a compound. My experience would indicate that a maximum speed of flow of gas over the work is usually restricted commercially by questions of expense and cost of circulation. In checking the maximum rate of flow indicated in this paper, I arrive at a rough figure of 60 inches per minute through the empty tube and, depending upon the size of the sample, this figure would, of course, be considerably increased by the test pieces. I believe that the more accurate indication of the effect of speed of the gases over the work might be found if the thermocouple were actually in contact with the test piece in the tube, so that the cooling effect of the gas on the surface of the work might be known. This is a very important consideration and determines in many commercial applications the maximum rate of flow of the carburizing gas.

I would be interested in a study in which circulation of the gases in the carburizing retort was obtained by a fan in the retort itself, so that instead of fresh gas being admitted at the rate indicated, the hot gases were recirculated to prevent stagnation of any gases in recesses, etc., in the work.

My experience would indicate that there is a maximum rate at which the surface austenite can absorb carbon at any given temperature and also a diffusion rate which is fixed by the temperature so that all we can hope to do by recirculation of gases, etc., is to approach this maximum uniformly at all parts of the exposed surface of the work.

Mr. Cowan's observation in regard to moisture checked with my experience as also does his conclusion in regard to scale upon the work to be carburized.

Those of us who know the extensive work which Mr. Cowan has done in the interest of gas carburizing and the great amount of time and effort he has given to develop the practical method of continuous gas carburizing, can appreciate that this short paper is a summary of a vast amount of work.

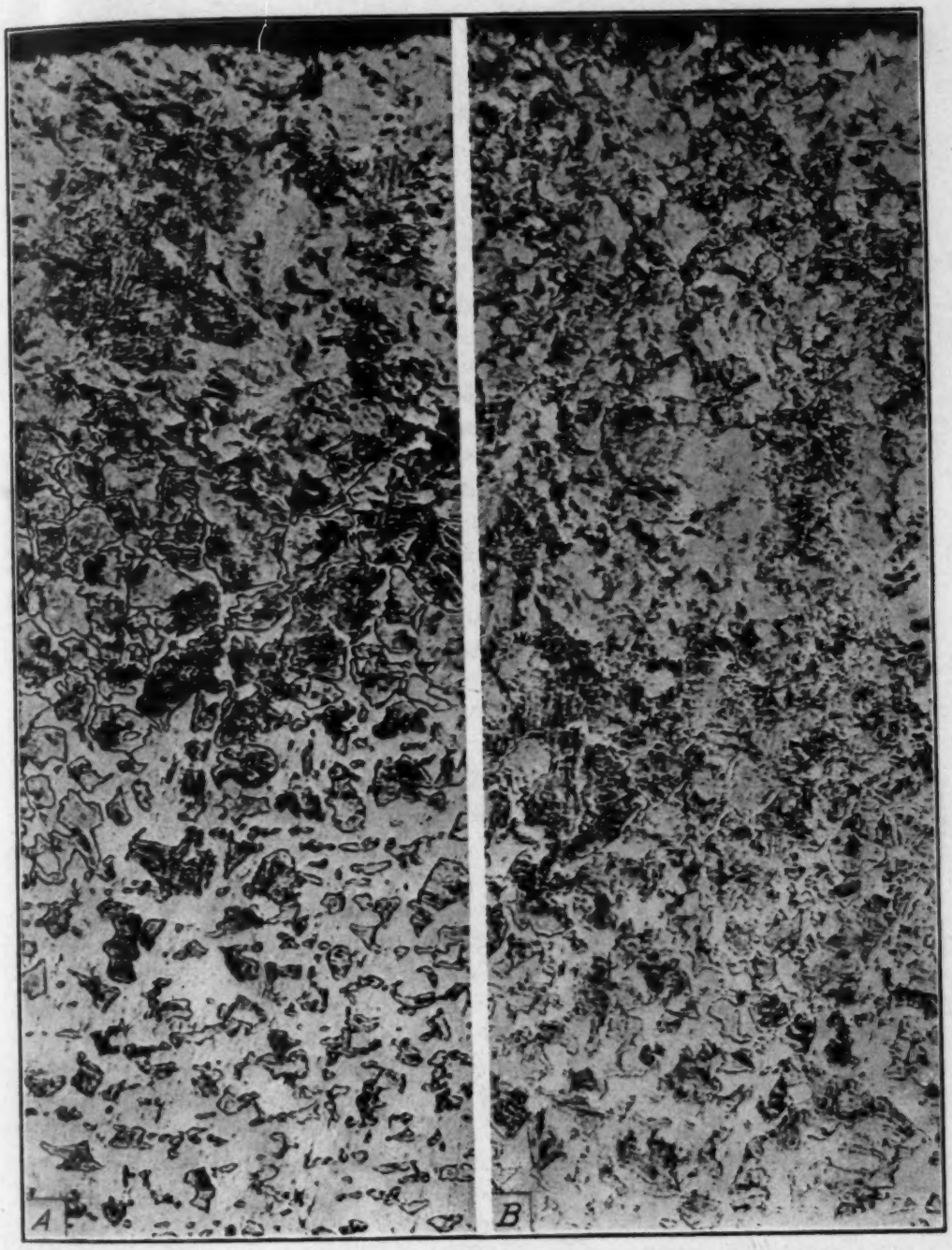
Oral Discussion

J. J. ROZNER:[2] This paper, on the effect of gas flow in continuous gas carburizing, is worthy of the author, R. J. Cowan, who has done much work on the subject.

It is noted that the experimental work has been done on S.A.E. 1020-90 steel samples, which is interesting to us, being a user of same. Our carburizing process is of the batch type, being done in a rotary carburizing furnace at 1750 degrees Fahr., under 15 pounds per square inch pressure, with city gas of the following approximate analysis:

	Per Cent
Carbon dioxide	0.8
Illuminants	2.1
Oxygen	0.2
Carbon monoxide	2.2
Hydrogen	25.7
Methane	57.0
Ethane	6.4
Nitrogen	5.6

[2]Metallurgist, Aetna Ball Bearing Mfg. Co.



S.A.E. 1015 Steel of Coarse Grain Type Carburized at 1750 Degrees Fahr. for 4½ Hours.

Fig. A—Machined Surface, Case Depth 0.039 Inch. × 100.
Fig. B—Mill Scale Surface, Case Depth 0.046 Inch. × 100.

In our production experience with this material, several variants entered into the carbon concentration control for a given case depth. The carbon content of the hypereutectoid section varied for the same flow of gas in this steel of different grain size, for banded and not banded steel, and also for steels

in which the silicon analysis checked 0.02 and below, the cause apparently being the slower diffusion rate of carbon due to these variants.

The matter of the gas channeling over the top of the work is also well indicated in rotary carburizing. Nonuniform case depth is obtained when pressure of the carburizing gas is below 10 pounds per square inch. This is due to a blanketing of the gas over the work without equal penetration through the mass being carburized.

Inasmuch as even gas distribution is obtained in rotary carburizers by increasing the pressure from 3 or 4 ounces to 10 pounds per square inch, might not the same condition be created in continuous carburizing by following the same practice? No doubt, the maintenance of pressure in the continuous process is of some problem—nevertheless, it would be interesting to note the effect of this, along with different volumes of gas passed over the work.

We wish to bear out a point that Mr. Cowan reports—namely, that of increased carbon penetration on surfaces covered with mill scale. In our work, we have both the mill scale surface of the Hemetite type, and machined surface on the part being carburized. The photomicrographs herewith illustrate this point. Case depth of the machined surface is 0.039 inch, while that of the mill scale surface, directly opposite, has a case depth of 0.046 inch. This specimen is S.A.E. X-1015 steel of the coarse grain type, and has been carburized at 1750 degrees Fahr. for 4½ hours.

Author's Closure

The discussions of this paper that have been submitted to the author are of the nature of contributions to the whole subject of gas carburizing. The importance of these is recognized and their presentation fully appreciated. For this reason, it is desired to consider each paper separately and prepare a reply to each that will deal with the points at issue in some detail.

My thanks are due Professor Daasch for calling attention to some very pertinent matters and for the chart he presents that describes some work of his own on the subject. This chart deals with the time of gas at temperature in relation to the case depth obtained. It suggests a method of analysis that may be quite helpful. To assist in making such analysis he requests some additional data on the tests I have reported. Since some of this information is to be found in the work of Bramley, attention is directed to the original reference. This reveals that Bramley used a glazed quartz tube 110 centimeters long with an internal diameter of 5.5 centimeters. Uniform temperatures were maintained over a "length of about 30 centimeters." In my own work, the specimens were passed through a tube 4 inches in diameter and 11 feet long in the hot zone as shown in Fig. 2. The gas flow, except as noted, was 11 cubic feet per hour. The temperature varied throughout the tube length but remained constant at the control points so that the specimens were above 1600 degrees Fahr. for 8 hours, the maximum temperature being 1750 degrees Fahr. for about 2.5 hours. The material dropped in temperature from 1600 to 1200 degrees Fahr. in 1 hour.

The statement in the paper as to the action of carbon dioxide upon carbon in the second step of the process is based upon the following considerations: In the heating zone the following reaction occurs: $2\,CO = C + CO_2$. The

CO_2 thus formed is not swept toward the outlet since the gas flow is not sufficiently rapid and the periodic opening and closing of muffle valves produces an agitation that carries these gases throughout the muffle. It is this movement of gases that makes CO_2 available in the second zone.

In regard to cooling the carburized specimens, it must be noted that the cooling chamber is not enclosed, therefore, when the work is removed from the high temperature area to the one cooled by air, the cooling is rather rapid. It is believed that the effect of this upon carbide diffusion would be but slight.

Further contributions from Professor Daasch will be looked forward to with interest. I hope the additional information will assist in developing the very interesting aspect of gas carburizing that he has suggested.

In the first part of Gordon T. Williams' discussion, he is looking toward a comprehensive theory of gas carburizing that will be both simple and satisfying. The outstanding fundamentals seem to embrace a carburizing agent and an oxidizing gas. The oxidizing gas serves to upset the equilibrium that tends to retard the carburizing rate. The usual oxidizing atmospheres are air, carbon dioxide and water vapor and these have been utilized in many ingenious ways. The suggestions made are very reasonable and valuable.

Mr. Williams deals in some detail with the effect of oxide scale upon the carburizing rate. The conclusions in all these cases are affected by a lack of exact information as to the conditions upon which they are based. For instance, if in the various tests under consideration there were always present a sufficiency of oxygen to give the maximum carburizing rate, it would seem that the presence of iron scale could have no further beneficial effect but, on the contrary, would be objectionable. However, most carburizing operations employ a large excess of carburizing gas in which case the scale would be beneficial. Such is probably the case in the tests I have reported. In the tests, Mr. Williams reports, there is probably a better balance between the carburizing and oxidizing gases so that the effect on the carburizing rate is not so noticeable, however, if the matter had been carried still further, there would have been a retardation similar to that obtained when machined holes are packed with mill scale and luted to retard carburization of these parts. It would seem, therefore, that the correlation of these varying results is to be found on such a basis as this. The broad statement in the paper must not be interpreted too broadly but only as applied to the tests described. Mr. Williams has done well to bring out this matter in his discussion and he has my sincere thanks.

We have, therefore, two explanations of this interesting phenomenon: One by Mr. Williams that relates it to carbon precipitation, and one by the author that connects it with a critical gas concentration. Both of these explanations seem reasonable; one of them may be right.

The remarks by Mr. Wyzalek are especially valuable because of his extended experience in the operation of continuous gas carburizing units and his well known studies covering all phases of the art. His remarks might well be considered addenda to my paper, supplying additional operating "Notes" that are extremely valuable.

The experience of Mr. Wyzalek goes beyond my own in his preference for propane, and I had not observed the ill effects of rust spots. His reference to cleaning is very much to the point and should be emphasized, being influenced

to some extent by the type of lubricant used. Some will distill off; others will bake on and interfere with results. The matter of gas distribution is of considerable importance, being determined by the character of work processed. These, and the other matters mentioned, are in line with good practice and I am grateful to Mr. Wyzalek for his valuable contribution.

The discussion by Mr. McQuaid, as would be expected, is both thought-provoking and suggestive. As to the effect of gas flow upon carburizing rate, it would seem that a fundamental consideration would be the removal of the gas film from the metal surface being carburized. To do this by mechanical means would require a great sweep of gas over the surface, the more rapid the better, until the limit is reached that is set by the rate of carbide diffusion referred to by Mr. McQuaid. In addition to this, we ought to consider the chemical means whereby reactions at metal surfaces are accelerated by the use of atmospheres in correct carburizing balance, that is to say, where the oxidizing agent present in the gas reacts with carbon at the film surface. It is believed that both these concepts may have a part in accelerating the reactions. Let us give the mechanical engineer a job but let us make the chemical engineer work also.

With regard to the recirculation of gases that have been used for carburizing, we are faced with the problem of liberating the carburizing potential of these gases. Circulation of itself is not sufficient, but an activating gas, usually containing oxygen, sets off the reaction and liberates the unused carburizing value of these gases. Again the problem is one of mechanics plus chemistry.

Some suggestions are made as to methods for conducting these tests. It would be valuable to know how the temperature of the work would be affected by gas flow. This might be done by attaching a thermocouple to the work; by determining the temperature of the effluent gases, or by exploring the reaction tube with a thermocouple mounted in a separate tube extending throughout the length of the reaction chamber. The latter method was used by Professor Bramley in the reference cited in the paper. It is hoped that someone will give us some more information on this matter. I am grateful to Mr. McQuaid for his stimulating discussion.

It is gratifying to note that Mr. Rozner's observations based on the operation of a rotary carburizing retort agree in some respects with those based on continuous carburizing. It is a fact that banded steels do not carburize normally. Grain size and chemical composition are also important. It is hard to account for gas channeling in a revolving retort filled with work. One would imagine that such tendencies would be smoothed out by the agitation. The effect of mill scale upon the work is very apparent from a study of the micrographs shown.

Without doubt an increase of pressure in the muffle of the continuous furnace would increase the carburizing rate, but such a method would encounter many operating difficulties. It is apparent that we cannot depend, in the continuous process, upon pressure to overcome stratification, but must rely upon viscous as distinguished from turbulent flow to obtain the best results.

The notes by Mr. Rozner, although springing from a different background of gas carburizing, bring out interesting points of similarity and difference, and point us to fundamental considerations for our analyses of these problems.

THE FRACTURE OF CARBON STEELS AT ELEVATED TEMPERATURES

By A. E. White, C. L. Clark and R. L. Wilson

Abstract

This paper presents results from stress versus time for rupture tests on electric-furnace and open-hearth carbon steels of the S. A. E. 1015 type at temperatures of 1000, 1200, 1300 and 1400 degrees Fahr. (535, 650, 705, 760 degrees Cent.) At 1000 degrees Fahr. fracture was obtained in periods varying from a few minutes up to 14,000 hours while at the higher temperatures maximum fracturing times of approximately 2000 hours were used.

The results indicate a straight-line logarithmic relationship to exist between stress and time for fracture at any given temperature provided oxidation or corrosion does not occur. If this does happen a break occurs and two lines rather than one represent the relationship.

The results likewise indicate the influence of time, temperature and stress on the resulting ductility and on the type fractures obtained. On the basis of these findings a means is proposed for determining the lowest temperature of recrystallization or equi-cohesive temperature.

CREEP tests, as normally conducted, have been criticized on the basis that they do not interpret completely the behavior of metals at elevated temperatures and neither do they yield the information desired by many users of materials for high pressure, high temperature conditions.

On the basis of the majority of creep tests, results are obtained showing the stress required for creep rates of 0.01 and 0.10 per cent per 1000 hours and through extrapolation the same stresses are oftentimes considered as applying to rates of 1.0 per cent in 100,000 and 10,000 hours, respectively. Since the specimens are seldom carried to actual fracture in the creep tests no information is obtained concerning the influence of stress, time, and temperature on the resulting ductility.

While certain applications require that the total deformation

A paper presented before the Eighteenth Annual Convention of the Society held in Cleveland, October 19 to 23, 1936. Of the authors, A. E. White and C. L. Clark are director and research engineer, respectively, Department of Engineering Research, University of Michigan, Ann Arbor, and R. L. Wilson is metallurgical engineer, The Timken Steel and Tube Co., Canton, Ohio. Manuscript received June 17, 1936.

shall not exceed definite small amounts during the life of the equipment, there are many others in which not only much greater deformations are permissible but also the life of the equipment is determined by a rather large total plastic deformation. In other words, in these latter cases the equipment is actually carried nearly to destruction and the expected life is relatively short.

Commercial applications of high temperature materials also differ in another respect. In certain instances, such as in turbine work, the normal operating temperature agrees closely with the maximum temperature to be encountered during normal service. In others, such as in cracking still tubes, temperatures considerably greater than the normal one may be encountered during certain periods of the operating cycle due to the insulating properties of the coke deposited on the bore of the tube. In applications of this type the major portion of the plastic deformation will occur during the time the tube is overheated and, furthermore, the rate of deformation during this period is, in some instances, considerably greater than that usually measured in the creep test.

On the basis of these facts it is believed that, while the standard creep testing procedure is satisfactory for those applications in which the permissible deformation is decidedly limited to small amounts, certain modifications in this procedure might be advisable, at least in a supplementary capacity, for those applications in which the parts are overheated, and therefore overstressed, thus producing deformation at relatively rapid rates.

Procedure

Believing, as previously discussed, that in certain applications the time required for fracture and the ductility characteristics up to fracture are of great importance, a series of tests were undertaken in which the time required for fracture was varied from a few minutes to hundreds of hours. The temperatures were likewise selected so as to represent a normal operating temperature of 1000 degrees Fahr. (535 degrees Cent.), as well as those from 1200 to 1400 degrees Fahr. (650-760 degrees Cent.) which would be encountered during the period the metal was overheated due to the deposition of some material, such as coke or scale.

From tests of this type stress-time for rupture curves were obtained and attempts were made to correlate these results with the short time tensile properties. In addition the fractures were exam-

ined metallographically and data were thus secured as to the influence of stress, time and temperature on the type fractures, that is, whether they were transcrystalline or intercrystalline. On the basis of these findings certain theories concerning creep were substantiated and a reliable means was obtained for determining the lowest temperature of recrystallization or equi-cohesive temperature.

The present paper is largely concerned with two plain carbon steels which differed mainly in that one was made in an electric-arc furnace while the other was an open-hearth product. Both were secured from The Timken Steel and Tube Co., in the form of hot-rolled annealed 1-inch bars. Their composition, grain size and hardness were as follows:

	Chemical Composition, Per Cent					Grain	Brinell
Type	C	Mn	Si	S	P	Size	Hardness
1015(OH)	0.15	0.46	0.28	0.021	0.019	5-6	111
1015(EL)	0.15	0.50	0.23	0.032	0.025	4-5	134

In certain cases the results obtained from these steels are compared with those previously reported by the authors[1] for an alloy steel of the chromium-silicon-molybdenum type. This is particularly true in the section dealing with a means of determining the lowest temperature of recrystallization.

In the stress-time for rupture tests the creep characteristics can also be determined if desired. This was done in certain cases but the present discussion will be largely confined to the rupture and ductility characteristics.

Results

The results obtained are grouped so as to show the influence of temperature and stress on the time required for fracture, on the type fractures obtained, and on the resulting ductility. Likewise a relationship between these results and those from the short-time tensile tests is discussed as is a means for determining the lowest temperature of recrystallization.

Stress Versus Time for Rupture. Both of the carbon steels were tested at temperatures of 1200, 1300 and 1400 degrees Fahr. (650, 705, 760 degrees Cent.) and the stresses were so selected that fracture resulted in time periods varying from a few minutes up to 2000 hours. The electric furnace carbon steel was also tested at 1000

[1] "Influence of Time on Creep of Steels," *Proceedings*, American Society for Testing Materials, Vol. 35, 1935, Pt. 2, p. 167.

degrees Fahr. (535 degrees Cent.) and in this case the maximum time for fracture was approximately 14,000 hours. The results obtained are given in Figs. 1, 2 and 3.

The resulting curves are, in all cases, similar in that they at first

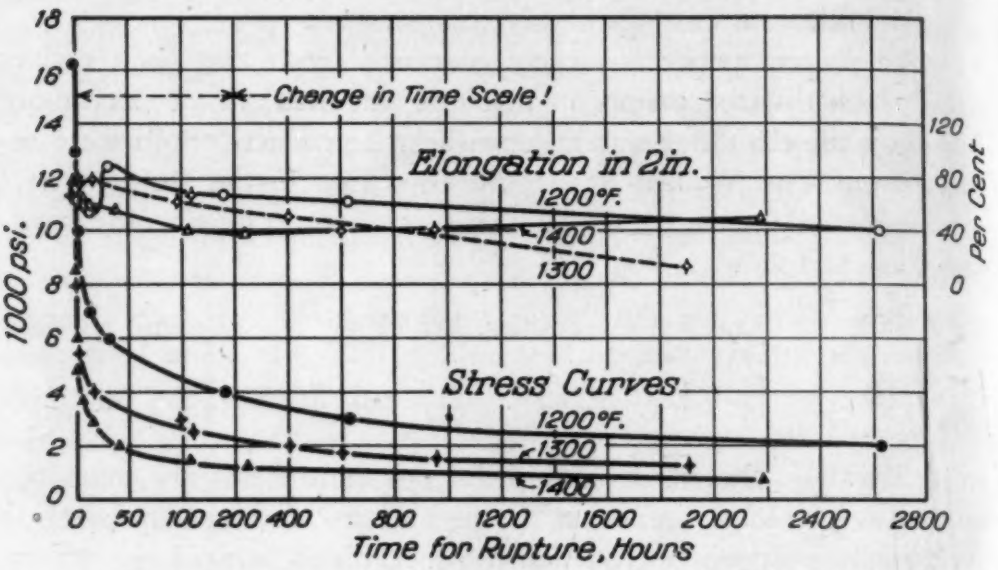


Fig. 1—Stress-Time for Rupture Curves for S.A.E. 1015 (OH) Steel at 1200, 1300 and 1400 Degrees Fahr.

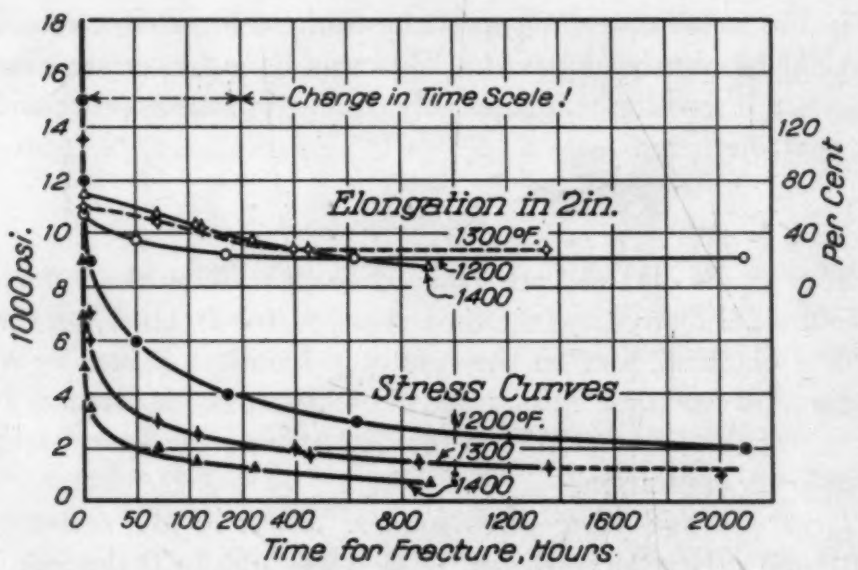


Fig. 2—Stress-Time for Rupture Curves for S.A.E. 1015 (EL) Steel at 1200, 1300 and 1400 Degrees Fahr.

drop rapidly as the time for fracture is extended and then tend to approach an asymptote. While the location of this asymptote is not definitely fixed the shape of the curves does indicate the stress below

which fracture would require very extended time periods. For example, from Fig. 3 it is apparent that at 1000 degrees Fahr. (535 degrees Cent.) a stress below 5000 pounds would require a very long period before actual failure occurred.

Curves of this type are believed to be valuable in those applications in which the equipment is overheated for relatively short-time

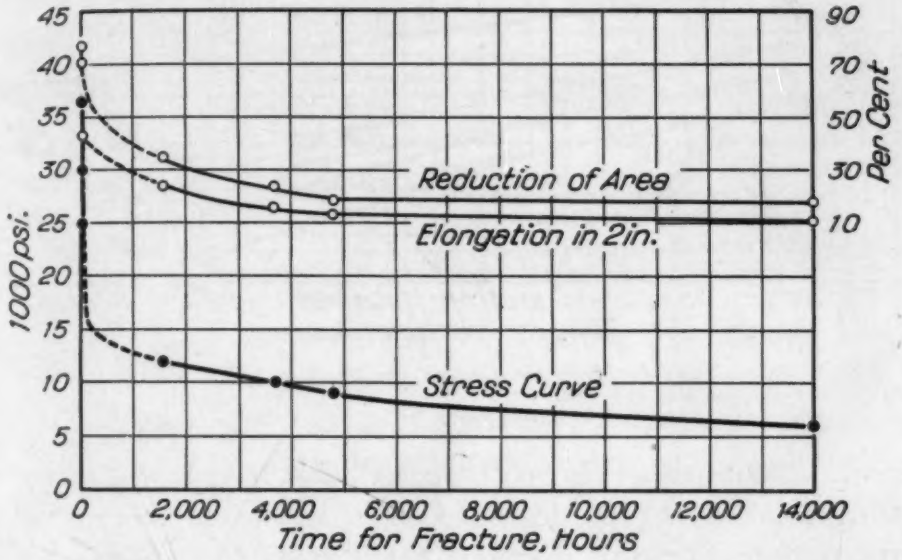


Fig. 3—Stress-Time for Rupture Curve at 1000 Degrees Fahr. for S.A.E. 1015 (EL) Steel.

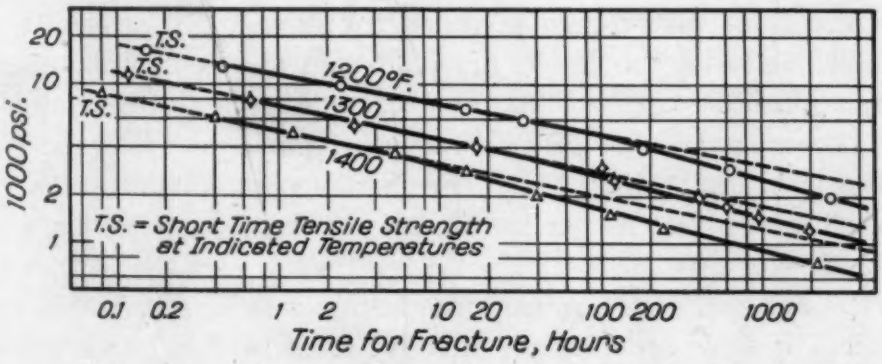


Fig. 4—Stress-Time for Rupture Curves for Steel S.A.E. 1015 (OH) at 1200, 1300 and 1400 Degrees Fahr.

periods and appreciable deformation is permissible in that they indicate very clearly the length of time this abnormal condition can be allowed to exist without causing undue damage. For purposes of illustration, if the normal operating temperature is 1000 degrees Fahr. (535 degrees Cent.) and unit stress 3000 pounds, then, insofar as actual fracture is concerned, the electric furnace carbon steel would

have indefinite life. If, however, the metal was overheated to 1200 degrees Fahr. (650 degrees Cent.) this stress would cause fracture in 630 hours; at 1300 degrees Fahr. (705 degrees Cent.), in 65 hours; and at 1400 degrees Fahr. (760 degrees Cent.), in only 10 hours. Therefore, this overheating should not exceed a few hundred

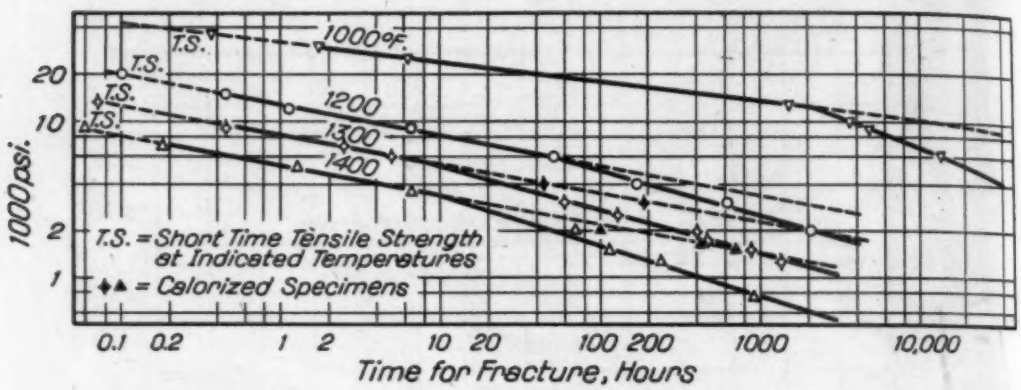


Fig. 5—Stress-Time for Rupture Curves for Steel S.A.E. 1015 (EL) at 1000, 1200, 1300 and 1400 Degrees Fahr.

hours at 1200 degrees Fahr. (650 degrees Cent.), only a few hours at 1300 degrees Fahr. (705 degrees Cent.), and in no case should the overheating temperature exceed 1300 degrees Fahr. (705 degrees Cent.) as a maximum.

Figs. 4 and 5 differ from those previously considered only in that logarithmic rather than cartesian coordinates are used. It will be noted that when the results are plotted in this manner a straight-line relationship exists for the shorter time period with a decided break occurring at time periods varying from 6 to 1500 hours depending upon the temperature. From this point on a straight-line relationship again exists but the slope is materially greater than that for the shorter time.

It is believed that a single straight-line relationship would exist regardless of time, provided the effective cross sectional area was not reduced by oxidation. This statement is substantiated by the fact that the time period required for this break in the relationship tends to vary inversely with the temperature. For example, with the electric furnace steel the time required for this change in slope was approximately 9 hours at 1300 and 1400 degrees Fahr. (705-760 degrees Cent.), 50 hours at 1200 degrees Fahr. (650 degrees Cent.) and 1500 hours at 1000 degrees Fahr. (535 degrees Cent.).

In order to further check this matter tests were conducted on

the electric furnace carbon steel at 1300 and 1400 degrees Fahr. (705-760 degrees Cent.) on specimens whose surfaces were protected from oxidation by calorizing. The results also shown in Fig. 5 definitely indicate that, in the absence of oxidation, a single straight-line relationship does exist for the time periods considered. Furthermore, it is believed that this relationship would also apply to much longer time periods, as is indicated by the results obtained at 1000 degrees Fahr. (535 degrees Cent.).

Rupture Tests Versus Tensile Properties. While many attempts have been made to correlate the results from the usual creep tests with those from the short-time tensile tests none have been completely successful. It is true that in certain cases a definite relationship is believed to exist between the proportional limit value and the creep characteristics but this applies only over a narrow temperature range and, furthermore, the location of this range varies with different metals. It is believed to be dependent on the lowest temperature of recrystallization or equi-cohesive temperature of the material.

It was, therefore, thought advisable to determine if a relationship might exist between the results from the time-rupture tests and those from the short-time tensile tests at the corresponding temperatures. Since in both of these tests the specimens are carried to actual fracture it was believed, that of the short-time tensile values, the tensile strength would offer the greatest possibility for this relationship. Accordingly, in Figs. 4 and 5, the lines were extended towards shorter time periods until they reached the stress corresponding to the short-time tensile strength, designated as TS on these curves.

The results obtained were encouraging in that they indicate approximately constant fracture times to be required for the stresses corresponding to the tensile strength. For example, with the open-hearth steel, Fig. 4, the fracture time for these stresses at 1200, 1300 and 1400 degrees Fahr. (650, 705, 760 degrees Cent.) were 0.15, 0.12 and 0.08 hours, respectively. Likewise, with the electric furnace steel the corresponding time periods were 0.10, 0.07 and 0.06 hours, while at 1000 degrees Fahr. (535 degrees Cent.) it was 0.37 hours. Furthermore, the relatively slight differences in these fracture time periods vary uniformly and are inversely proportional to the temperature. With both steels the minimum time was required at the higher temperature and the maximum at the lowest one.

The usefulness of this relationship is obvious. First, it indicates that a metal's ability to resist fracture at these more elevated tempera-

tures is proportional to the tensile strength values at the corresponding temperatures. Thus, for those applications in which overheating occurs during portions of the operating cycle, the short-time tensile strength may give a good indication of the behavior under these abnormal conditions. It may, at least, serve as a yardstick in sorting materials of the same general type.

This relationship is also valuable from a second standpoint in that by knowing the tensile strength as well as the stresses required to produce fracture in two relatively short-time periods the ideal stress versus time for fracture curve can be reproduced for indefinite periods. In order to convert this into an actual working curve it would be necessary to ascertain the oxidation and corrosion resistance of the material and also the influence of these factors on the strength characteristics.

It is, of course, unwise to draw too broad conclusions from the limited results but the findings obtained do indicate this relationship to exist for the two carbon steels considered. Furthermore, preliminary results obtained from certain pearlitic alloy steels over this same temperature range also substantiate the present findings.

The ability of the tensile strength to prophesy the time for fracture should not be considered as too rigid a relationship. Even though the fracture time for the tensile strength is approximately constant the slight variations that do exist are sufficient to appreciably influence the results because of the slope of the logarithmic lines. For example, two steels having the same tensile strength at a given temperature may require appreciably different stresses to produce fracture in a given time if the tensile strength causes fracture in one case in 0.10 hours and in the other case in either 0.08 or 0.12 hours.

Time Versus Resulting Ductility. As pointed out earlier the ductility a metal possesses up to fracture may be of great importance particularly in those applications in which overheating occurs. In many cases the life of the part is based on a relatively large plastic deformation and if failure should occur without appreciable deformation no warning of impending failure would be given and thus serious damage might result.

Tests of the type herein considered are of value in this respect for since the specimens are carried to actual failure the ductility characteristics and especially the elongation can be determined. Reduction in area values are, in many cases, not easily obtained at the higher temperatures due to the rather severe oxidation.

The elongation of each of the fractured specimens was determined and the results are shown graphically in the upper curves of Figs. 1, 2, and 3. In all cases there is a tendency for the ductility to decrease as the time required for fracture is extended. With the open-hearth carbon steel the minimum value did not fall appreciably below 40 per cent at either 1200 or 1400 degrees Fahr. (650-760 degrees Cent.). At 1300 degrees Fahr. (705 degrees Cent.) the condition was more serious in that after 1900 hours the elongation was only 12 per cent. Furthermore, the slope of the curve indicates that there would be an additional sharp decrease as the fracture time was extended further.

The results obtained from the electric furnace steel are similar although in this case temperatures of 1200 and 1300 degrees Fahr. (650, 705 degrees Cent.) did not seriously influence the ductility, the minimum values being of the order of 20 per cent. The decrease at 1400 degrees Fahr. (760 degrees Cent.), however, is more pronounced as after 900 hours a value of only 16 per cent resulted.

The results obtained from this steel at 1000 degrees Fahr. (535 degrees Cent.) Fig. 3, are of interest. Whereas the short-time tensile specimens possessed an elongation of 42.5 per cent and a reduction of area value of 77.0 per cent, the corresponding values for the specimen which required the maximum time for fracture, approximately 14,000 hours, were only 11 and 18 per cent, respectively. This represents a very large decrease in the ductility characteristics and indicates that the material is being embrittled due to the combined action of stress and time at this temperature. As has already been pointed out by the authors[2] the cause of this embrittlement may be attributed to the pronounced intergranular oxidation that occurred during the test even though the oxidizing condition was not severe.

This intergranular type of oxidation was not observed in either of the carbon steels at the higher temperatures. This difference is believed to be due to the fact that, while the oxidation resistance of the grain boundary is less than that of the crystal proper, the rate of oxidation at the higher temperatures is so great that the slight difference in the resistance of the two phases is not brought into play. On the other hand, at 1000 degrees Fahr. (535 degrees Cent.) the attack of the mass is relatively slight and thus this preferential difference is of great importance.

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[2]"Influence of Time at 1000 Degrees Fahr. on the Characteristics of Carbon Steel," *Proceedings*, American Society for Testing Materials, Vol. 36, Pt. II, 1936, p. 139.

The observed decrease in ductility as the fracture time is extended may be due, in part at least, to the oxidation of the specimen. If this is true then it is evident that the intergranular type of attack produces a much greater decrease than when the oxidation is more general. The influence of oxidation and corrosion on the resulting ductility is a subject of great importance and one worthy of consideration.

Time Versus Type Fracture. If certain of the theories which have been advanced concerning the creep phenomena are correct then the type fracture obtained should vary depending upon the temperature and the time required for fracture. According to one theory the fracture should be transcrystalline at temperatures below the lowest temperature of recrystallization, regardless of the time required for fracture, while at temperatures above, it should be transcrystalline for the more rapid rates of testing and intercrystalline when sufficient time is allowed.

The fractures of these specimens were examined metallographically and certain of the typical microstructures are shown in Figs. 6 to 9 inclusive. At all four temperatures the short-time tensile specimen possessed a severely deformed structure and the fracture was largely transcrystalline. At 1200 degrees Fahr. (650 degrees Cent.) and above recrystallization has started in the tensile specimen and the resulting grain size varies directly with the temperature.

Likewise the specimens requiring the maximum period of time for fracture all possessed the same type structure in that it was equiaxed and thus strain-free and fracture was largely intercrystalline. At the higher temperatures appreciable grain growth has occurred and the resulting grain size is again proportional to the temperature for a given time.

The specimens requiring the intermediate time period for fracture possessed structures in the process of change from the strained condition of the short-time tensile specimen to the unstrained and equi-axed state of the extended-time specimens. As would be expected the rate of this change is more rapid the higher the temperature.

On the basis of these findings it is apparent that 1000 degrees Fahr. (535 degrees Cent.) is above the lowest temperature of recrystallization of the electric furnace carbon steel. Otherwise the structure would have been deformed after the extended test as no means would have existed for removing the strain produced. Also,

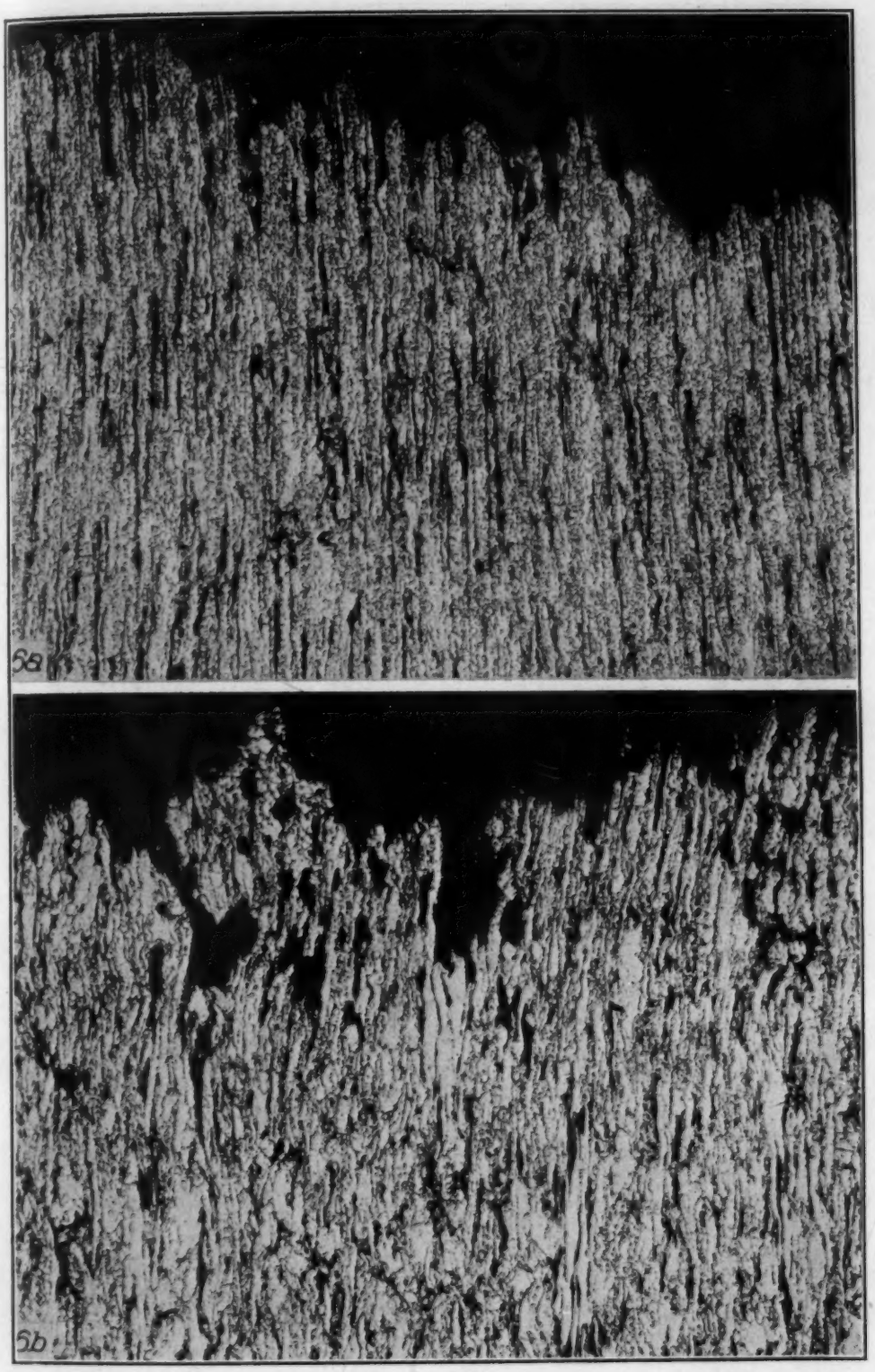


Fig. 6 a and b—Influence of Time and Stress on the Fracture of Open-Hearth S.A.E. 1015 Steel at 1200 Degrees Fahr. a—Short-Time Tensile Fracture. b—7000 Pound Stress, 14.2 Hours for Rupture.

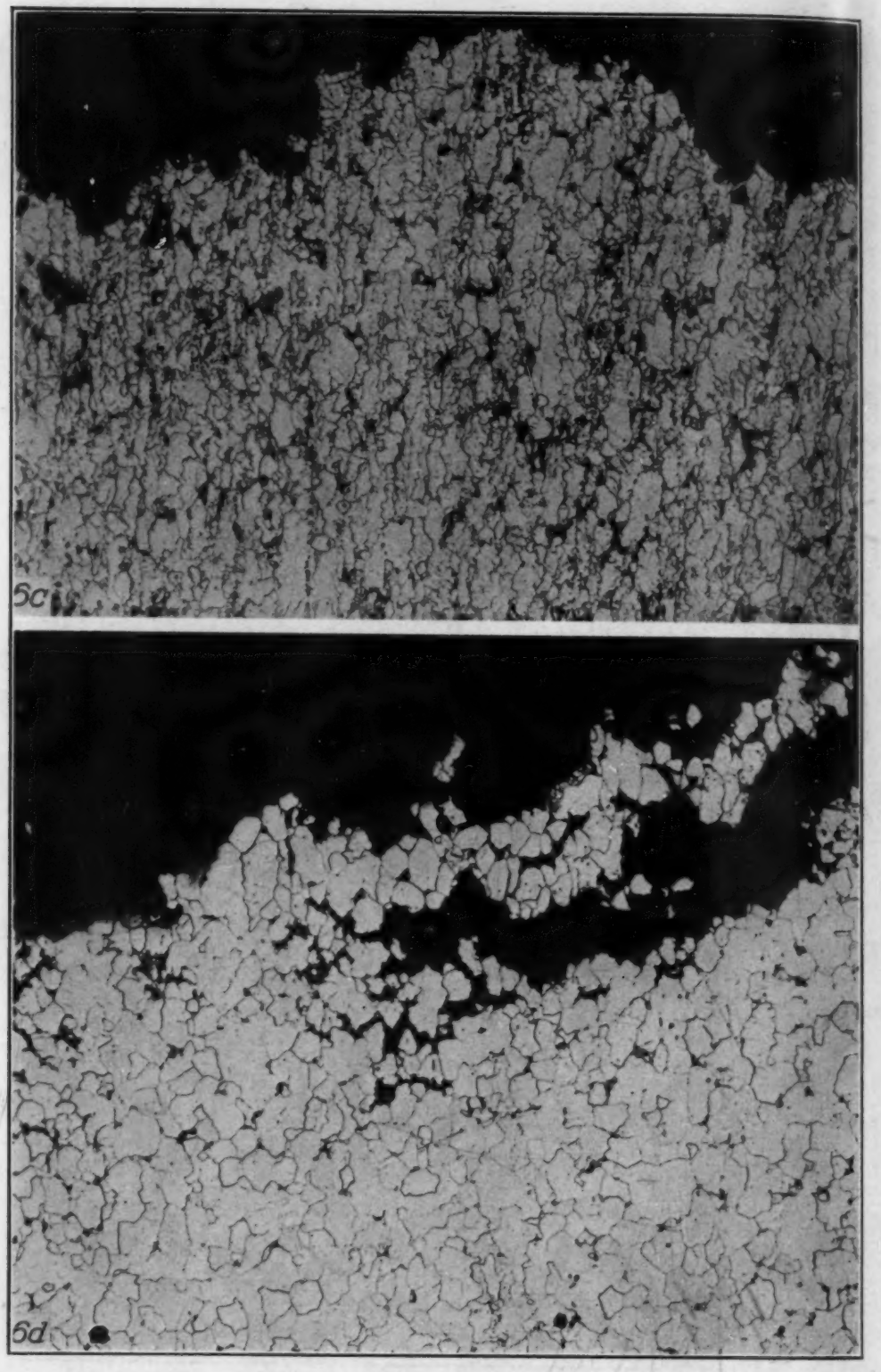


Fig. 6 c and d—Influence of Time and Stress on the Fracture of Open-Hearth S.A.E. 1015 Steel at 1200 Degrees Fahr. c—4000 Pound Stress, 180 Hours for Rupture. d—2000 Pound Stress, 2626 Hours for Rupture.

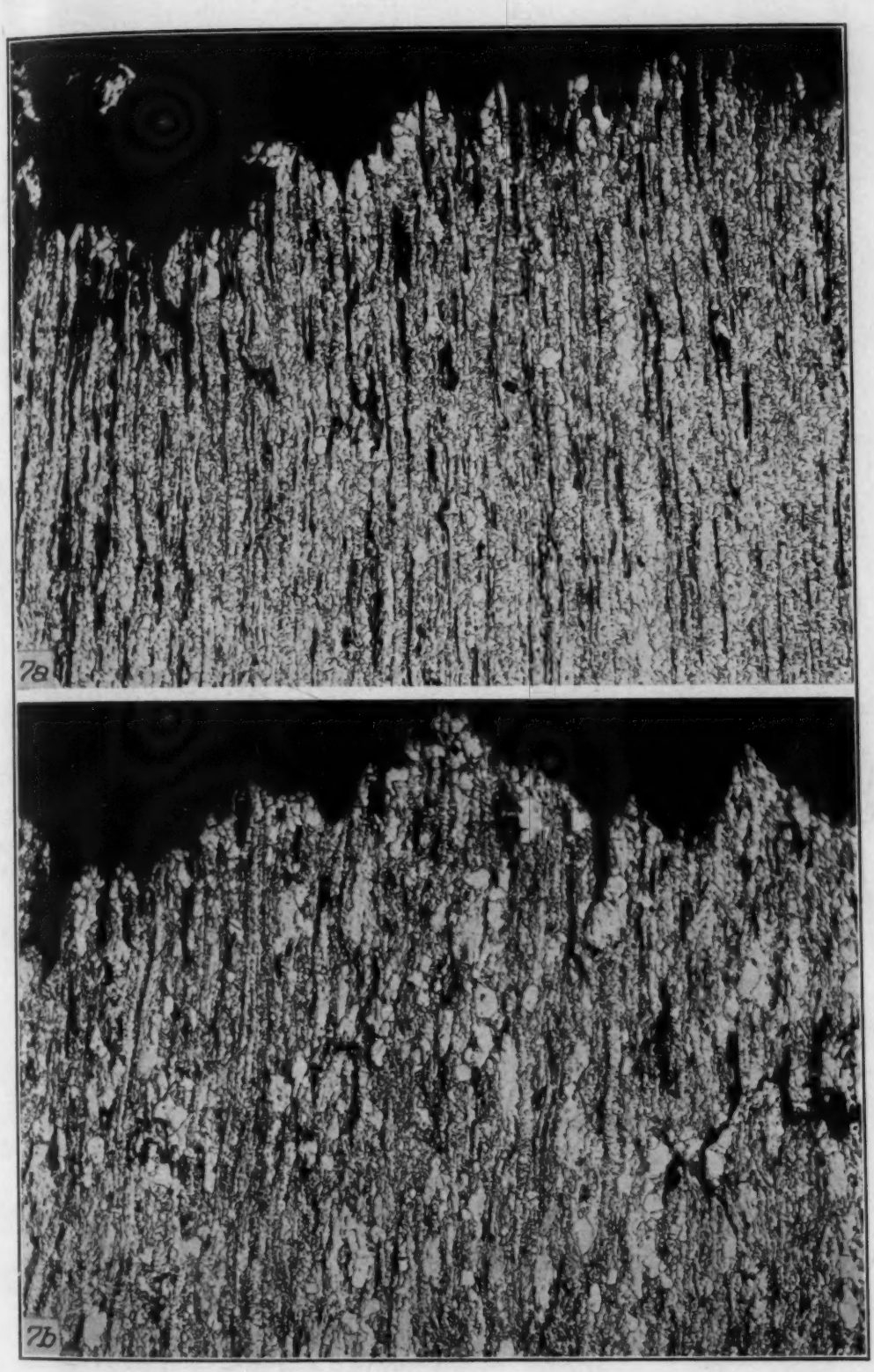


Fig. 7 a and b—Influence of Time and Stress on the Fracture of Open-Hearth S.A.E. 1015 Steel at 1300 Degrees Fahr. a—Short-Time Tensile Fracture. b—5500 Pound Stress, 2.90 Hours for Rupture.

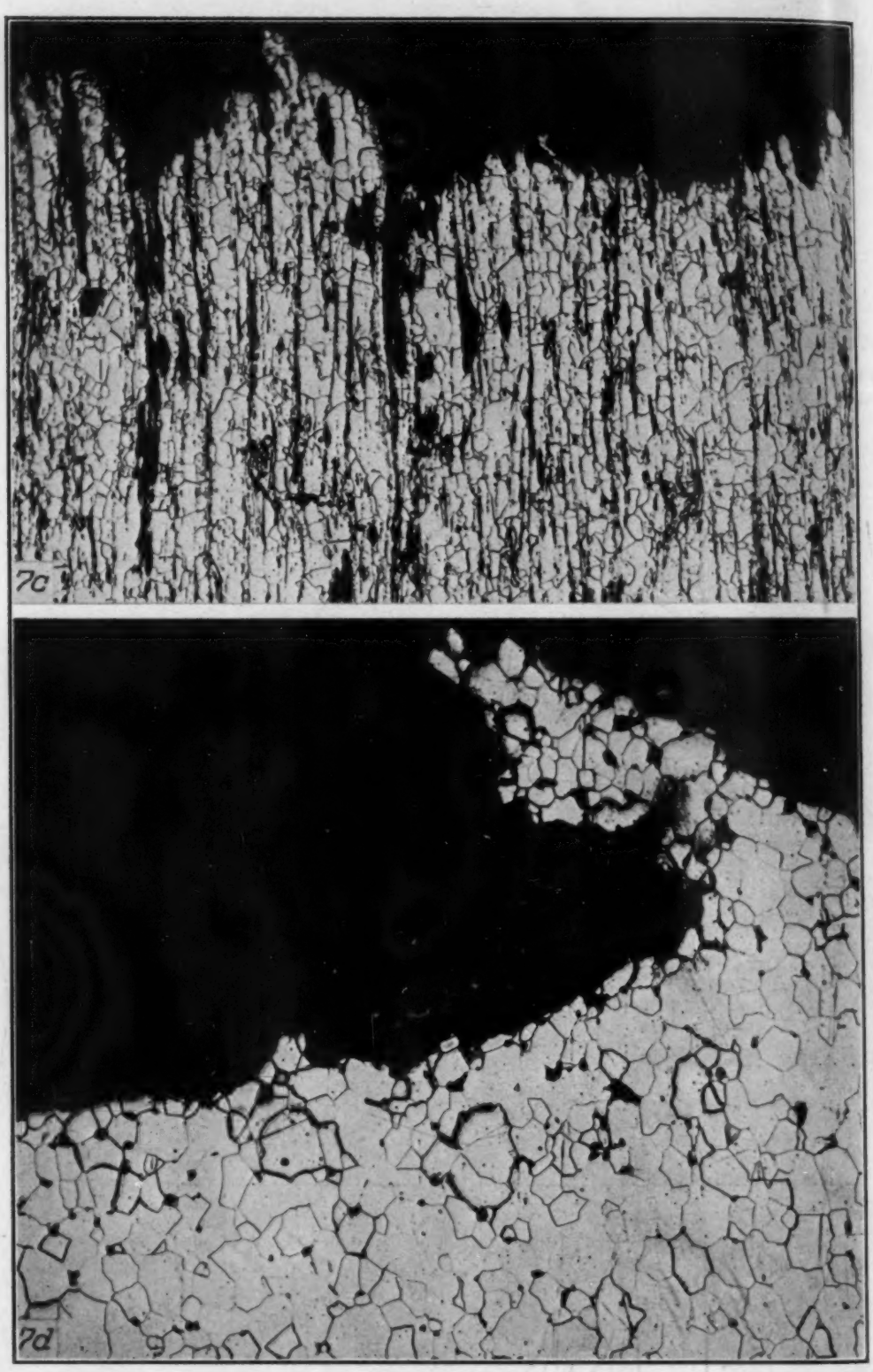


Fig. 7 c and d—Influence of Time and Stress on the Fracture of Open-Hearth S.A.E. 1015 Steel at 1300 Degrees Fahr. c—400 Pound Stress, 16.48 Hours for Rupture. d—1500 Pound Stress, 953 Hours for Rupture.

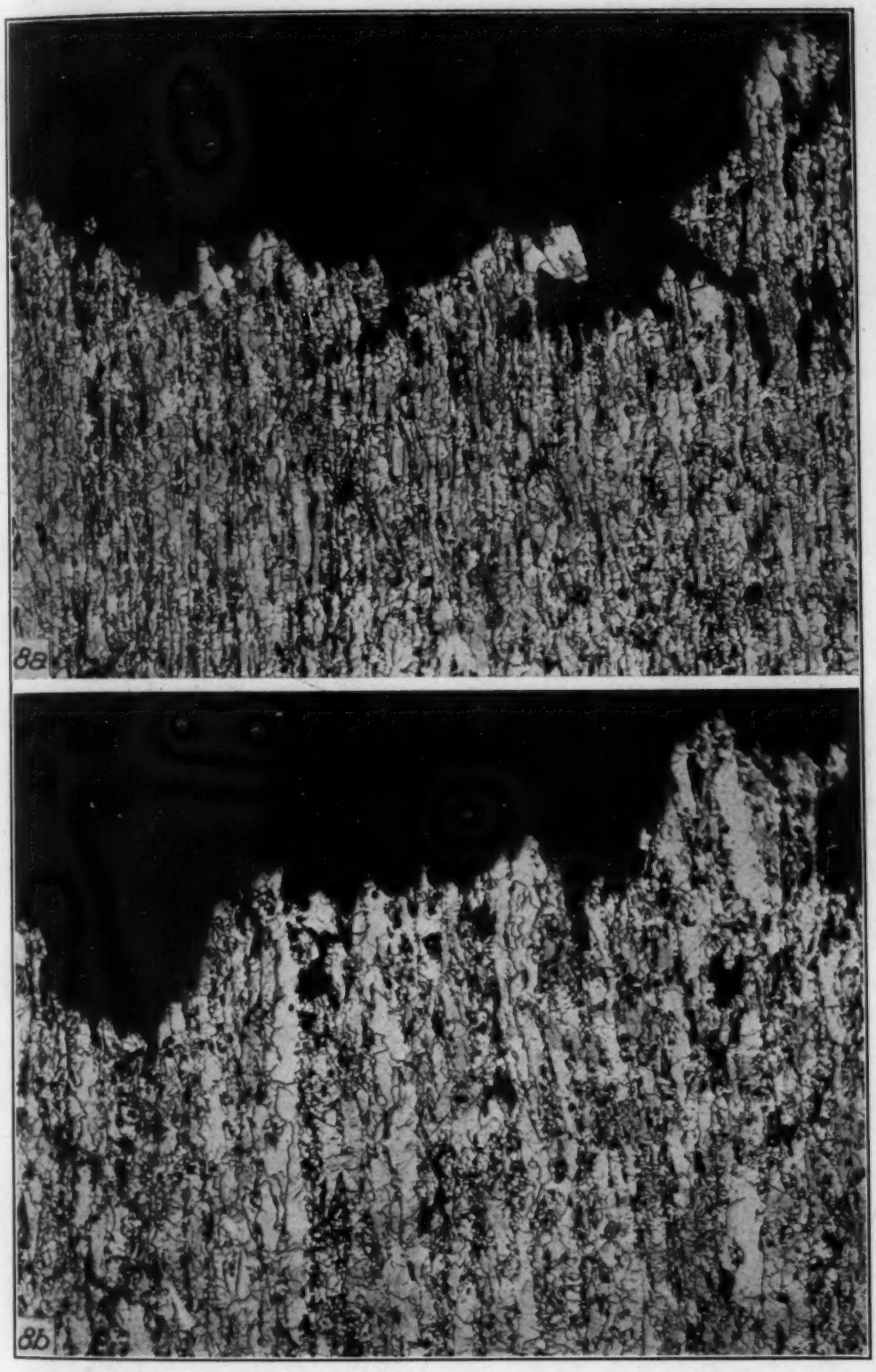


Fig. 8 a and b—Influence of Time and Stress on the Fracture of Open-Hearth S.A.E. Steel at 1400 Degrees Fahr. a—Short-Time Tensile Fracture. b—3700 Pound Stress, 5.23 Hours for Rupture.

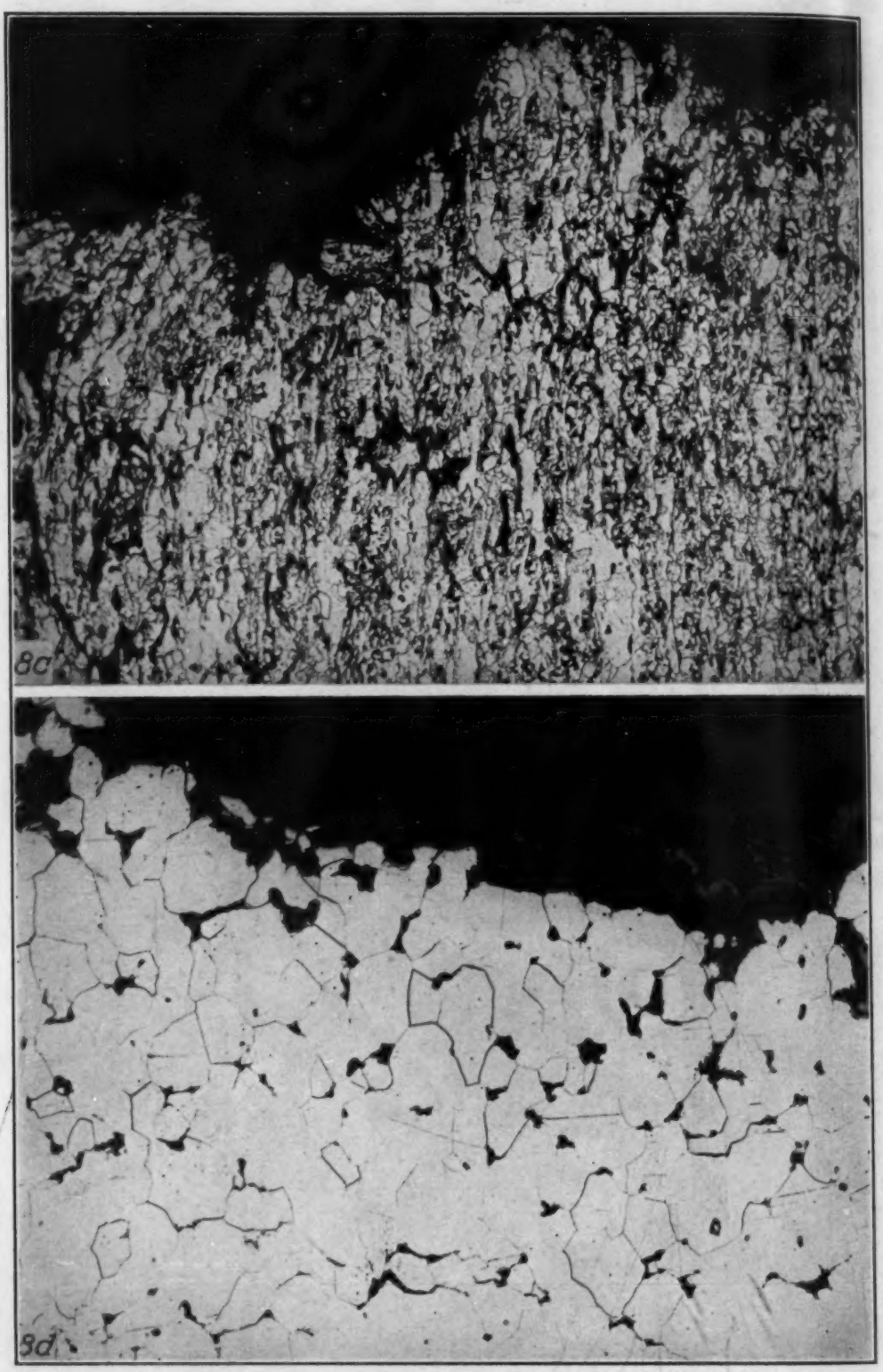


Fig. 8 c and d—Influence of Time and Stress on the Fracture of Open-Hearth S.A.E. 1015 Steel at 1400 Degrees Fahr. c—2850 Pound Stress, 14.77 Hours for Rupture. d—750 Pound Stress, 2180 Hours for Rupture.

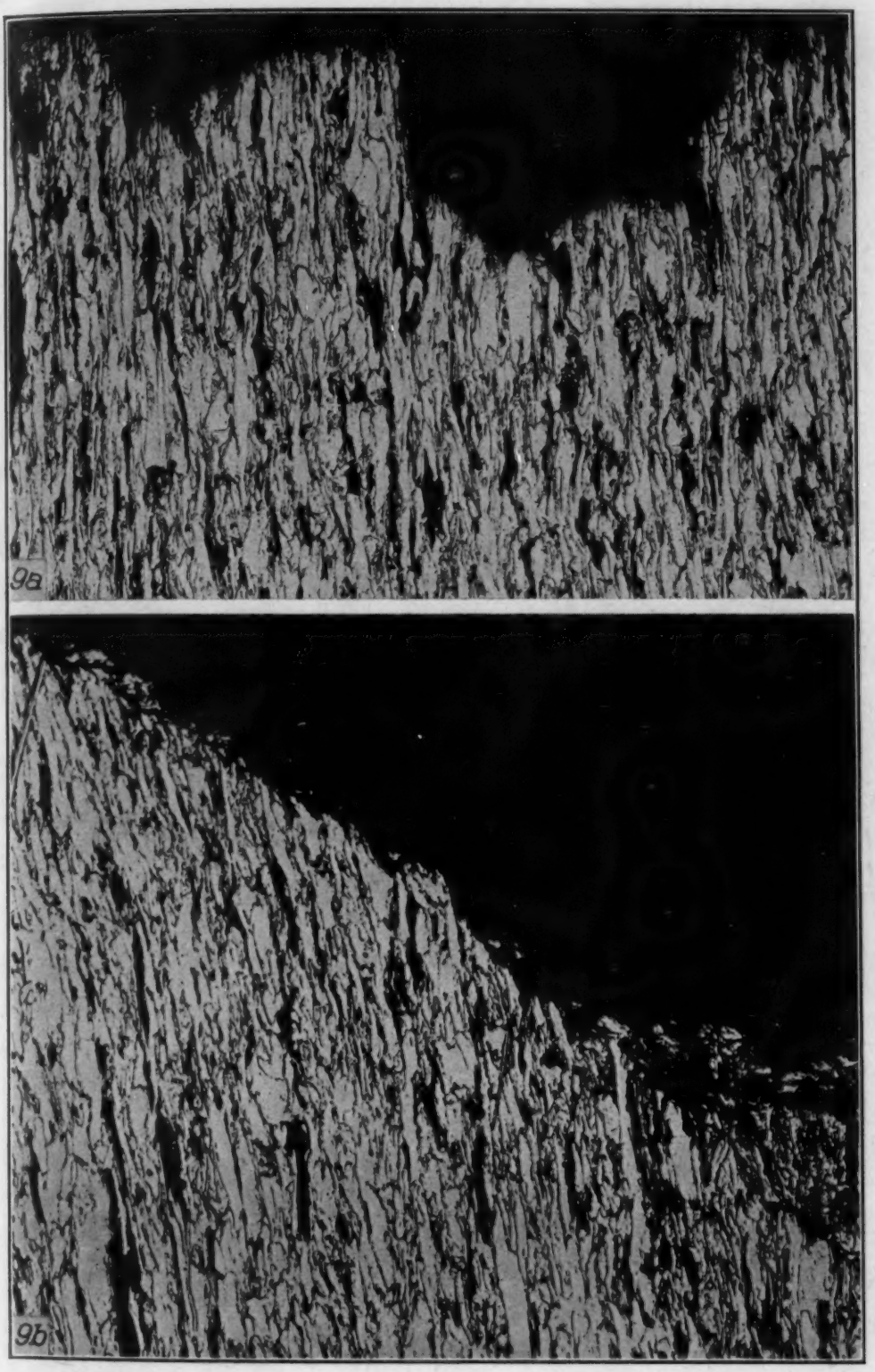


Fig. 9 a and b—Influence of Time and Stress on the Fracture of Electric Furnace S.A.E. 1015 Steel at 1000 Degrees Fahr. a—Short-Time Tensile Fracture. b—25,000 Pound Stress, 6.4 Hours for Rupture.

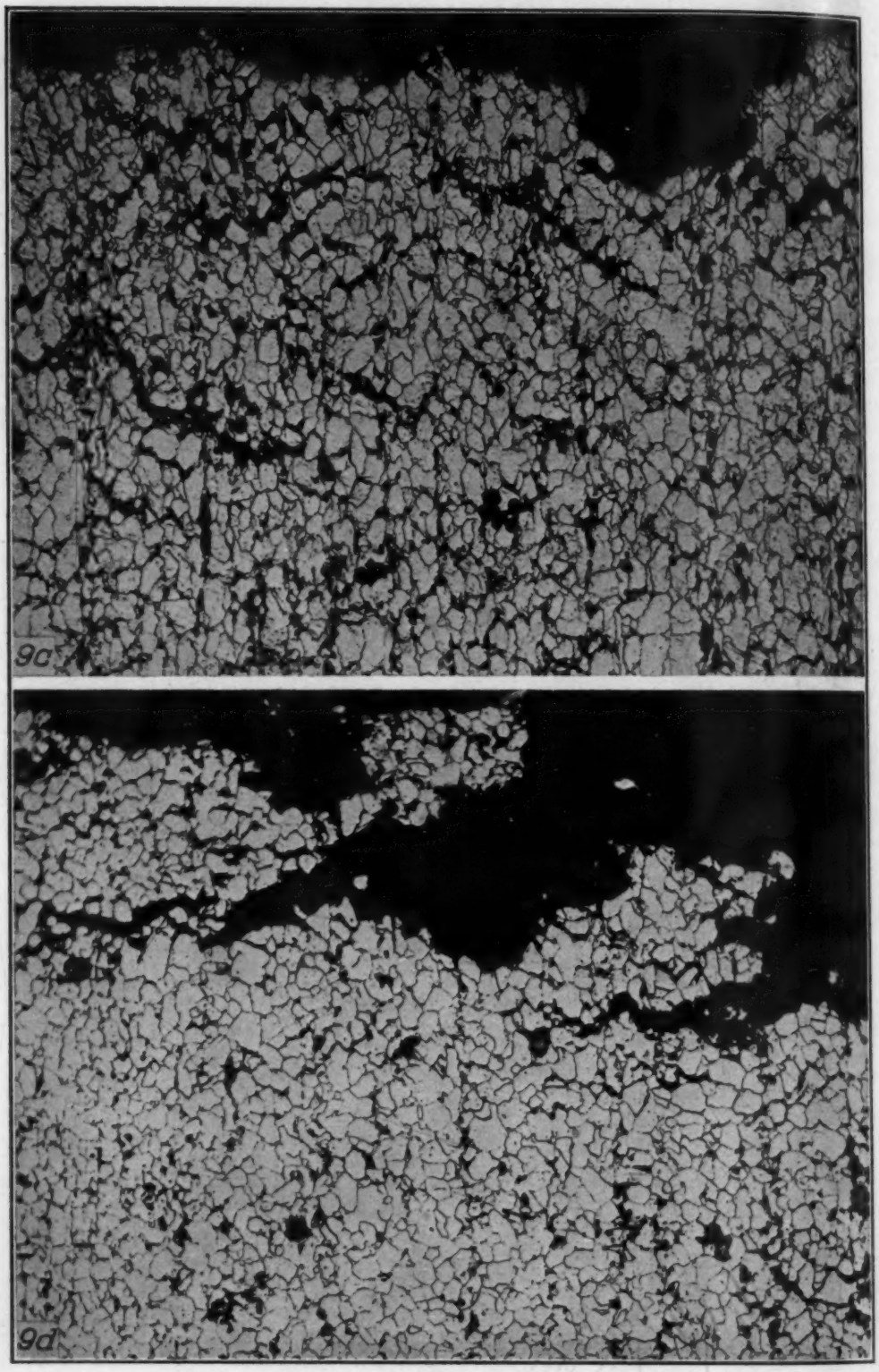


Fig. 9 c and d—Influence of Time and Stress on the Fracture of Electric Furnace S.A.E. 1015 Steel at 1000 Degrees Fahr. c—12,000 Pound Stress, 1552 Hours for Rupture. d—6000 Pound Stress, 13,950.75 Hours for Rupture.

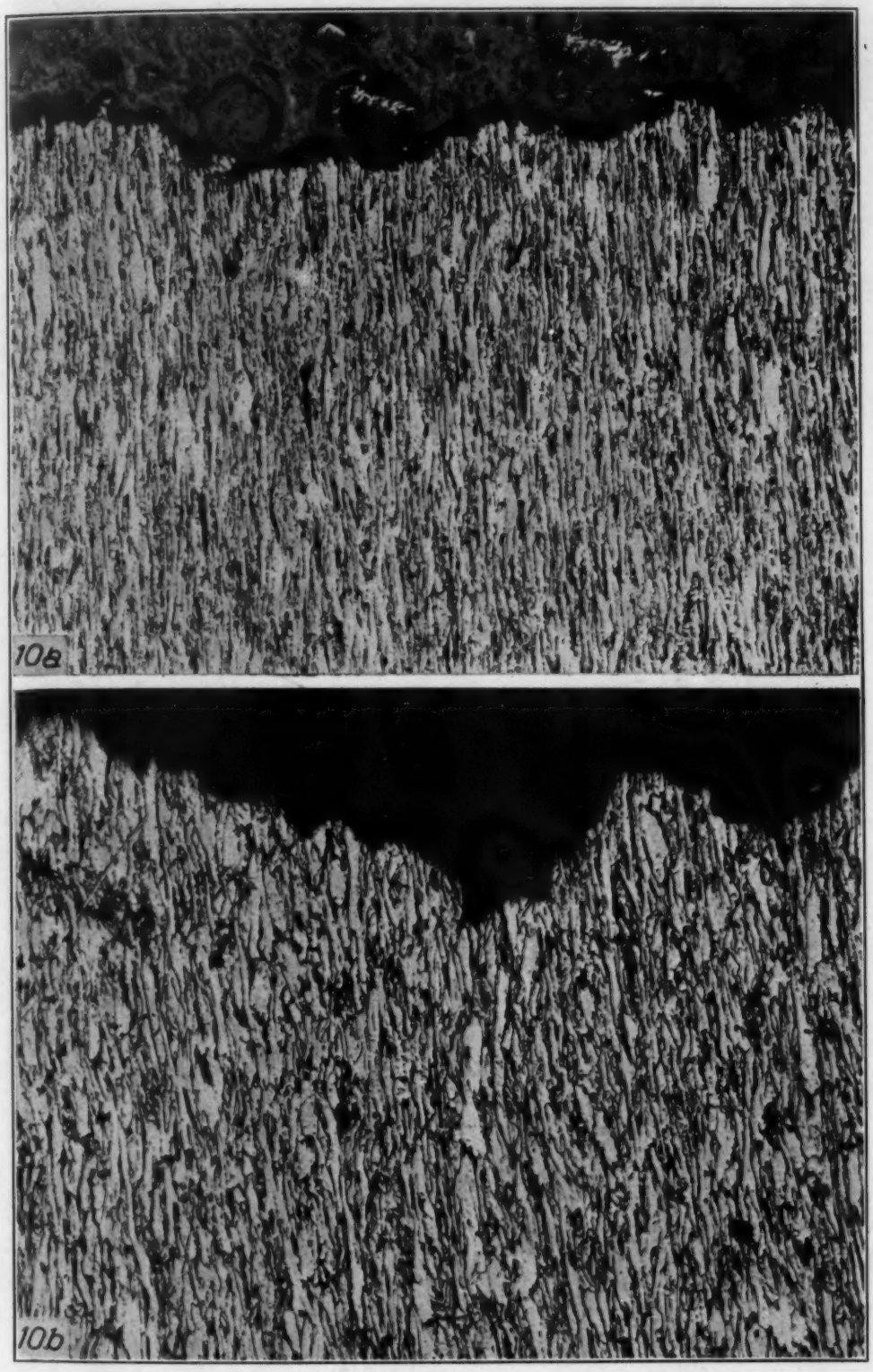


Fig. 10 a and b—Influence of Time and Stress on the Fracture of Cr-Si-Mo Steel at 1000 Degrees Fahr. a—Short-Time Tensile Fracture. × 100. b—34,100 Pound Stress, 2625.5 Hours for Rupture. × 100.

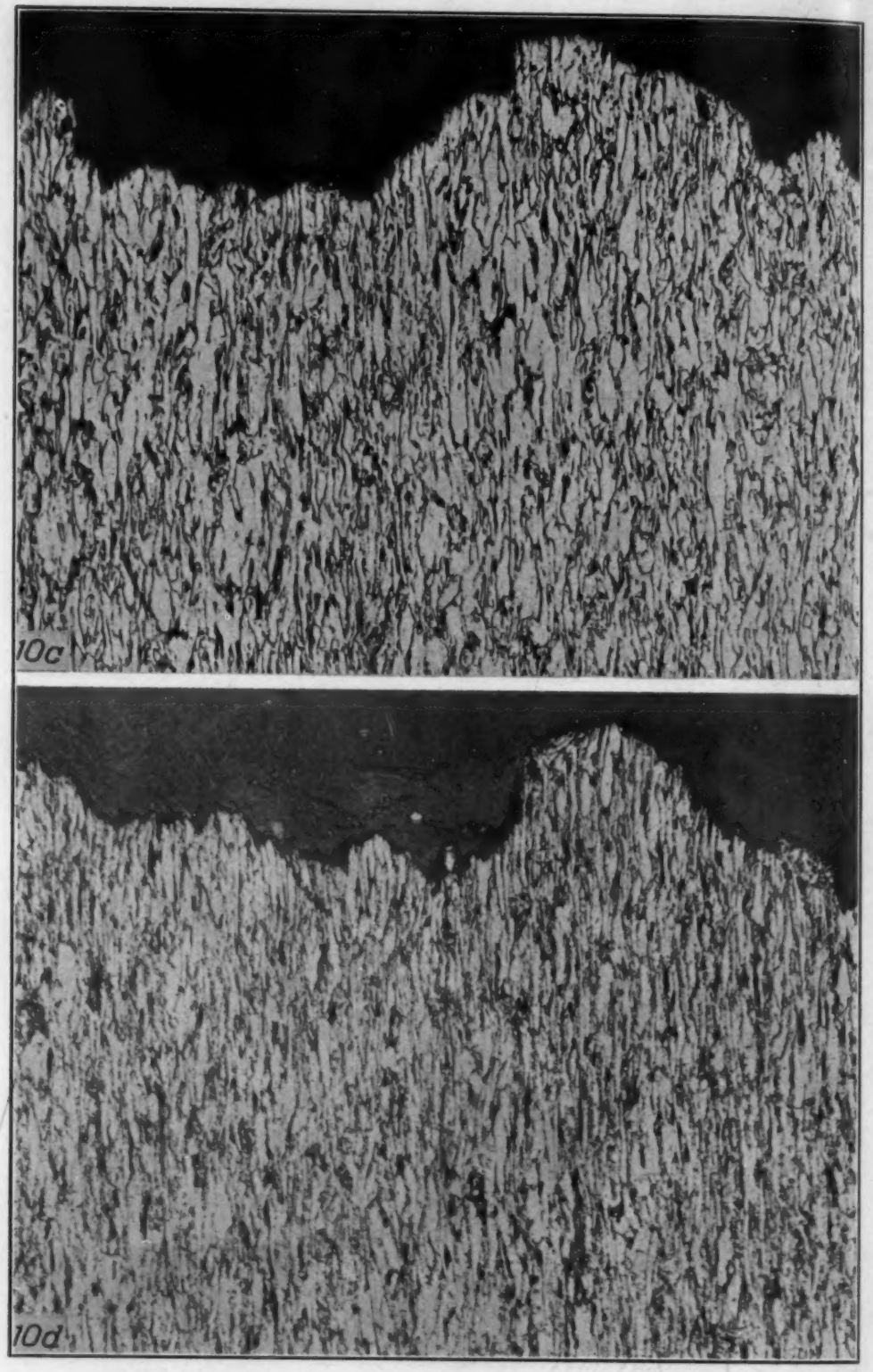


Fig. 10 c and d—Influence of Time and Stress on the Fracture of Cr-Si-Mo Steel at 1000 Degrees Fahr. c—30,000 Pound Stress, 3250 Hours for Rupture. × 100. d—24,600 Pound Stress, 6151 Hours for Rupture. × 100.

it is believed that Fig. 9 substantiates the belief several times expressed that the lowest temperature of recrystallization and the equi-cohesive temperature are one and the same. When the structure is strained the fracture is largely transcrystalline, while when it is unstrained it is largely intercrystalline.

These results, as well as certain of those previously presented, also furnish proof to the fact that the creep characteristics are greatly influenced by the position of the test temperature with respect to the equi-cohesive temperature. It has been shown, for example, that the creep resistance of a certain chromium-silicon-molybdenum steel at 1000 degrees Fahr. (535 degrees Cent.) is from four to five times that of a plain carbon steel. On the basis of the structures given in Fig. 9, 1000 degrees Fahr. (535 degrees Cent.) is above the equi-cohesive temperature of the carbon steel. On the other hand, the fractures of the alloy steel, shown in Fig. 10, clearly indicate that the equi-cohesive temperature of this material is above 1000 degrees Fahr.

Determination of Equi-Cohesive Temperature. The results obtained also indicate a means for determining the equi-cohesive temperature or lowest temperature of recrystallization of metals. Specimens of the metal in question can be fractured at varying time periods and the structures examined. Even though the material is such that the path of fracture cannot be accurately followed the condition of the structure will indicate whether the test temperature is above or below the desired one. If, for example, the grains are severely deformed, as in Fig. 10, it can be concluded that the test temperature is below the lowest temperature of recrystallization and thus the equi-cohesive temperature. If, however, the grains are equi-axed after extended time periods, as in Figs. 6 through 9, then the test temperature is above the equi-cohesive temperature. Furthermore, a careful examination of the fractures for time periods up to 300 or 400 hours will provide sufficient information for the purpose.

Hardness tests on the fractured specimens are likewise valuable in this connection. If the hardness at the extended time fracture is greater than that of the original material, then strain-hardening has been produced. If, on the other hand, it is equal to or less than that of the original material no strain-hardening is present.

Conclusions

The results obtained from the stress-time for rupture tests permit the following conclusions:

1. The stress-time for rupture curves indicate the maximum time a metal can be overheated under any given stress and also the maximum temperature to which it can be overheated. They also indicate the maximum allowable stress that can be employed for prolonged periods without causing actual failure. They do not, however, produce information as to the rate of deformation.

2. The short-time tensile strength at the more elevated temperatures may serve as an indication of the resistance to fracture of a metal at the corresponding temperature.

3. The short-time tensile strength together with the stresses required for fracture in two relatively short-time periods, such as 2 hours and 10 hours, will establish the ideal stress-time for rupture curve for indefinite periods. Before the actual curve can be obtained, however, information must be available concerning the resistance to oxidation and corrosion.

4. The ductility characteristics of the fractured specimens indicate the influence of time, temperature and stress on the maximum deformation up to fracture and thus yield valuable information both as to the stability of the material and the amount of deformation to be expected before failure occurs.

5. Under rapid testing the structure will be deformed and the fracture transcrystalline regardless of the test temperature. For prolonged testing times the same condition will result if the test temperature is below the lowest temperature of recrystallization, but if it is above, the structures will be equi-axed and the fracture intercrystalline.

6. The examination of the fractured specimens, therefore, affords a good means for determining the equi-cohesive temperature or lowest temperature of recrystallization.

Acknowledgments

The authors desire to express their appreciation to The Timken Steel and Tube Company for their financial assistance which has made these investigations possible and for their permission to publish the findings. Particularly do they wish to express their gratitude to F. J. Griffiths, H. H. Timken, Jr., and K. B. Bowman.

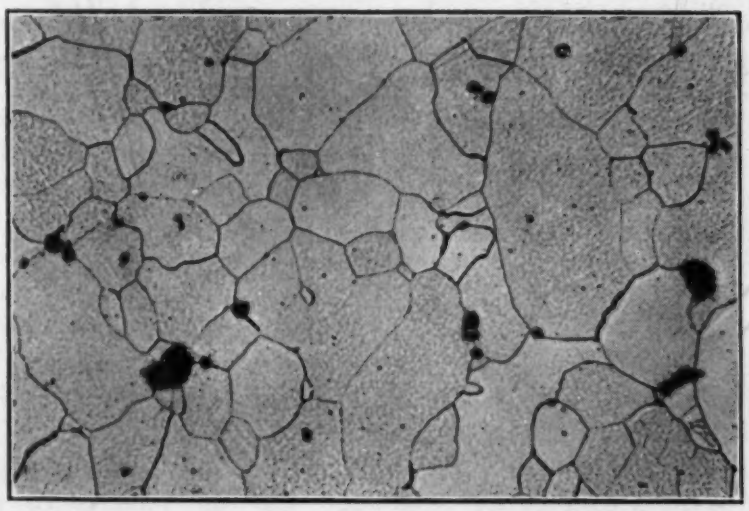
DISCUSSION

Written Discussion: By R. W. Moore, Socony-Vacuum Oil Co., New York City.

While investigating several still tube failures from one of our cracking units during the past five years, we have acquired some data and information which we thought might be of interest to present in connection with the present paper.

The tubes in question which have failed are open-hearth of the S.A.E. 1015 type steel, and have been in service for varying periods of time up to 5½ years, the latter time representing about 45,000 hours of service. The furnace temperature is 1450 degrees Fahr. The temperature of the oil going into the tubes is about 700 degrees Fahr. The oil temperature at the outlet is about 1030 degrees Fahr. Allowing for a temperature gradient of the oil through the bank of tubes as well as a further temperature gradient between the oil in the tubes and the furnace temperature, it would be our estimate that the probable temperature of the metal has been between 1000 and 1100 degrees Fahr. The pressure of the oil entering the tube bank is about 650 pounds per square inch. We do not know what the pressure gradient is through the tube bank.

It is of interest to note that the metal of these tubes has been almost completely graphitized. The pearlite has passed through the spheroidized stage, the carbides have migrated to the grain boundaries and have further broken up with the formation of nodular graphite. The microstructure is shown below. The subject of graphitization in low carbon steel has been presented in a paper by Kinzel and Moore.[1]



The physical tests of the metal as taken from one of the tubes after about 45,000 hours of service are herewith given.

Ultimate Tensile Strength	39600 pounds per square inch
Elongation in 2 inches	44.0 per cent
Reduction in area	75.0 per cent
Rockwell "B" hardness	26-30

[1] *Metals Technology*, American Institute of Mining and Metallurgical Engineers, Technical Publication No. 565, September 1934.

We note that Messrs. Clark and White in a previous paper given at this convention on the creep characteristics of metals, page 843,[2] have pointed out that there is a question as to whether speroidization and migration of the carbides to the grain boundary represent the condition of maximum stability. We believe the above represents at least one practical answer to the question.

It would appear from the authors' photomicrographs on pages 874—Fig. 6d, 876—Fig. 7d, 878—Fig. 8d, and 880—Fig. 9d, that the carbides are absent and that a condition such as that described above has resulted. We should like to ask the authors if this is the case.

Written Discussion: By A. C. Chamberlin, Bethlehem Steel Co.

The ultimate object of all high temperature testing is to provide reliable information on which to base decisions concerning choice of steel heat treatment and design calculations for a given application. To this end the authors have provided data on a relatively rapid method for determining the preferred heat treatment to be used for a given steel when subjected to known operating temperatures.

An examination of the structures at the point of fracture of short time high temperature tensile specimens has been used in the past only as a means of providing interesting information on the character of the fracture, but probably never for the purpose of indicating the optimum heat treatment for a steel. Information of this kind is interesting in the light of service experience which has not been in general agreement with laboratory creep observations. It has been proven that some alloys having only moderately good creep properties may give an unusually good account of themselves when placed in severe high temperature service. Conversely, other alloys, exhibiting excellent creep properties in the laboratory, may not be as satisfactory in service.

With the above in mind, any test which can be used to augment the conventional long time creep test may assist in a better understanding of that test, to the end that a more satisfactory agreement may be secured between actual operating behavior and laboratory creep tests.

Oral Discussion

H. F. Moore:[3] Similar results to those reported by the authors for metals under high temperature have been found at the University of Illinois by Mr. B. B. Betty and myself for lead and lead alloys at ordinary room temperatures. Specimens of lead under test in a testing machine will fail at (say) 2000 pounds per square inch and the test will consume approximately 20 minutes. Specimens from the same sheet of lead will fail under a steady load of approximately 800 pounds per square inch in about a month. On a semi-log. diagram with stress as ordinates (ordinary coordinates) and time as abscissas (log. coordinates) the relation between tensile strength and time required for fracture is not far from a straight line for times up to two or three months.

M. Gensamer:[4] I would like to comment on the authors' remarks concerning the equi-cohesive temperature. As I understand the concept of the equi-cohesive temperature, there is some temperature above which a metal of a given structure will fail by the propagation of an intercrystalline crack, and

[2]C. L. Clark and A. E. White, "Creep Characteristics of Metals," Transactions, American Society for Metals, Vol. 24, 1936, p. 843.

[3]Professor of Engineering Materials, University of Illinois, Urbana, Ill.

[4]Carnegie Institute of Technology, Pittsburgh.

below which this same structure will fail by a transcrystalline fracture. Now in the paper to which we have just listened these two types of fracture were produced in different grain structures; the intercrystalline fractures correspond to equi-axed grains, the transcrystalline fractures correspond to elongated cold-worked grains.

The authors have nicely demonstrated the effect of structure or plastic deformation on the nature of the fracture, but I cannot see that they have measured or even demonstrated the existence of an equi-cohesive temperature.

R. F. MILLER:[5] Does the microstructure of the fracture of tubes which have failed in service below and above the "equi-cohesive" temperature bear out the observations made on steels fractured in tensile creep tests?

Observations made by myself at the Hammond Laboratory, Yale University, during the past two years have disclosed that slow extension in zinc, aluminum, and silver single crystals takes place by movement on crystallographic glide planes specified by previous investigators for abundant plastic deformation. It might therefore be expected that in steel showing cold work and transcrystalline fracture, such as that shown in Fig. 6a, page 873, some degree of preferred orientation might be encountered. I would like to inquire whether Dr. Clark has investigated this subject.

The tensile tests on aluminum single crystals at Hammond Laboratory also disclosed a marked change in manner and rate of extension below and above the recrystallization or equi-cohesive temperature.

Authors' Closure

Mr. R. W. Moore's statements about the physical condition of S.A.E. 1015 steel after 45,000 hours of service at high temperature in an oil cracking furnace are of especial importance because the literature does not contain many references to work of this kind. The authors have not examined the microstructure of carbon steel after such a long period in commercial service, but no graphite was found in a tensile specimen subjected to 4000 pounds per square inch unit stress for 20,200 hours at 1000 degrees Fahr. Extreme spheroidization was noted, however, and the carbide in the microstructure was not easily detected at low magnification as it seemed to lie in plates along the grain boundaries. This probably accounts for the difficulty of seeing the carbide in the photomicrographs to which Mr. Moore has referred in the discussion.

Long time at a temperature beneath the carbon change-point of a steel is the main factor in converting iron carbide into graphite and iron. If additional data on this subject were available, the conditions governing the rate of decomposition of the carbide could be learned. Besides, it would be useful to know the effect of various alloying elements on the stability of the carbides formed in the special steels that have been recommended for service at high temperatures.

In answer to Mr. Miller's question about the microstructure of tubes that have failed in service, we may state that both plain carbon and alloy steel tubes showed intergranular fractures associated with a microstructure consisting of equi-axed grains and coalesced carbide when failure had occurred at temperatures above the equi-cohesive temperature. There has been no opportunity as

[5]U. S. Steel Corp., Research Laboratory, Kearny, N. J.

yet for the study of service failures of tubes that have occurred at temperatures below the equi-cohesive temperature, as examples of this kind are rare. Tubes usually fail due to overheating rather than to excessive bursting pressure at a predetermined operating temperature.

X-ray examinations have not been attempted for the specific purpose of finding the degree of preferred orientation of crystals in steel showing cold work and transcrystalline fracture. Such tests as have been undertaken were carried out simply to detect the presence of strain in pieces that had fractured after some thousands of hours under stress at 1000 degrees Fahr. Very probably the amount of cold work necessary to establish a preferred orientation would be greater than this. The authors think Mr. Miller's remarks about the deformation of tensile tests on single crystals of aluminum bear directly on the subject under discussion. For either single crystals or aggregates of crystals the rate of extension would increase when the self-annealing tendency would overbalance the strain-hardening.

Mr. Gensamer's comments serve as a reminder that the equi-cohesive temperature of steel is a controversial matter. The interpretation will of course depend upon the definition of the term. If it can be accepted, as the authors believe, that the lowest temperature or recrystallization of a steel after strain-hardening is the same thing as the lowest temperature of equi-cohesion, then it would not be possible to have an intergranular fracture except in a micro-structure having equi-axed grains. Otherwise, distorted and strained crystals would be admitted to exist about the lowest temperature of recrystallization after strain-hardening, which would be an inconsistency.

The concept of an equi-cohesive temperature has been useful in dealing with many situations arising in research work on the strength of steels at elevated temperatures. Whenever the facts seem to fit the case, attention is drawn to evidence that has been found to support the postulated existence of an equi-cohesive temperature, although observations of this nature have not been offered as proofs that there is an equi-cohesive temperature.

Professor Moore's description of the rupture of lead under steady tensile load is most welcome, as the behavior of this metal at ordinary temperatures resembles that of steel at elevated temperatures. Following Professor Moore's suggestion, some of the data from which several of the straight-line graphs were obtained when using logarithmic co-ordinates were replotted to semi-logarithmic co-ordinates in order to see whether the relationship between breaking stress and time for rupture could be expressed better by this method. It was found that plotting the data to semi-logarithmic co-ordinates gave a straight-line curve for longer periods of breaking as, for example, between one hour and 1000 hours. On the other hand the curve showed a changing steep slope for the breaking times under one hour. Since the present work features an attempt to use the short time high temperature tensile strength of steel for design purposes in the absence of other more reliable strength data, it might seem preferable to retain the log-log method of plotting as a means of correlating the short time and the long time breaking strength of steels.

The authors agree with Mr. Chamberlin that the long time rupture test may become useful to supplement the conventional creep test.

AN IMPROVED METHOD FOR DETERMINING GAS CONTENT OF MOLTEN STEEL

A Rapid Control Method for Analysis of FeO in Molten Steel

By Weston Hare, Leland Peterson and Gilbert Soler

Abstract

A method is described for analyzing the gas content and accurately calculating the FeO in molten steel at any stage of the working of the heat.

The apparatus used is described and the method of sampling, analyzing and calculating gas content or FeO content is outlined.

Typical analyses are given for per cent FeO and per cent H_2 at various points through the working of electric and open-hearth heats.

Introduction

An accurate study of the gases present in molten steel and of those evolved from commercially produced steel during solidification has been, until recently, a difficult task due to the general inability of sampling these gases without contamination by air or without serious loss of a part of the evolved gases. An early means of studying the gases evolved from steel ingots during freezing was by means of a bell-shaped sampling jar which was allowed to freeze in the top of the ingot, the evolved gases being drawn off by a vacuum pump. In 1920, E. Piwowarsky studied the gases evolved from ingots by this method[1] in a determination of the effect of time of silicon addition on the gassiness of the ingot in the mold. He found that the evolved gases consisted at first largely of carbon monoxide (CO) with less hydrogen (H_2), carbon dioxide (CO_2) and methane (CH_4), but at later stages of freezing and down to 700 degrees Cent. (1290 degrees Fahr.) became preponderantly nitrogen (N_2). Hibbard in 1921 wrote a summary of the knowledge of gases in steel as then known.[2] He lists the gases present in molten steel as H_2, CO, N_2, CO_2, and CH_4 in order of decreasing prevalency.

[1] E. Piwowarsky, *Stahl und Eisen*, Vol. 40, 1920, p. 773.

[2] H. D. Hibbard, *Blast Furnace and Steel Plant*, Vol. 9, 1921, p. 51, 139, 193.

A paper by Weston Hare, research engineer, Leland Peterson, research assistant and Gilbert Soler, manager of research, Steel and Tube Division, The Timken Roller Bearing Co., Canton, Ohio. Manuscript received June 1, 1937.

It is fairly certain that except perhaps in the case of hydrogen, whose atomic size is small, all gases present in steel are there as compounds of iron or other metallic constituents. Thus, there is presumably no free oxygen, carbon monoxide, or nitrogen present in steel, but iron oxides, iron nitrides, etc. The true solubility of most gases in a metal is probably practically zero. The carbon monoxide which represents a very large percentage of evolved gases from the solidifying metal is doubtless produced by the reaction

$$FeO + C \rightarrow CO + Fe$$

the CO being evolved as rapidly as produced, barring possible supersaturation under exceptional conditions.

In 1927 Wüster and Piwowarsky published a paper[3] in which the present work was in principle preempted, although we developed our method without knowledge of their prior work. They used an apparatus for drawing molten steel into an evacuated chamber in which the gases evolved were collected and measured. Their apparatus for this study was rather cumbersome and inconvenient, however, so that apparently little work except that of a laboratory nature was accomplished. They found that the gases from molten steel consisted mainly of CO, H_2 and N_2 in roughly equal volumes, with less but apparently excessive amounts of CH_4 and CO_2 in addition.

Ameen and Willners report some work[4] in which they have studied gases evolved during freezing in a special mold which permitted collection of gases. This work is available to us only in abstract form, but they state that the gases from molten steel are largely CO and hydrogen, with only small amounts of nitrogen, carbon dioxide, etc., a conclusion in agreement with our present work.

Gas analysis has also frequently been used in connection with the study of samples of steel taken from ingots or rolled steel products. The method of attack discussed in this paper, however, relates to the gathering of vital information as to the character of the steel before it leaves the furnace, enabling the manufacturer to correct and improve his product instead of having to rely on tests made on the finished bar after solidification has caused a loss of much of the gaseous products.

Development of Sampling Method

When the study of gases in molten steel was undertaken in our plant in 1934, we had two primary objectives in view: first, to ascer-

[3] A. Wüster and E. Piwowarsky, *Stahl und Eisen*, Vol. 47, 1927, p. 698.
[4] E. Ameen and H. Willners, *Jernkontorets Annaler*, Vol. 110, 1926, p. 107-24.

tain the nature and amounts of various gases present in our alloy steels at various stages during the working of the heats and teeming, and second, to develop a method of using the evolved CO as a quantitative measure of the degree of oxidation (per cent of FeO) at various stages of the heat for research purposes and for actual production control. A considerable degree of success has been attained in both phases of the work.

Sampling consists in general of immersing one end of a steel sampling tube, previously evacuated, in a test spoonful of molten steel. The portion of the tube immersed in the steel is closed with a very thin steel or copper diaphragm which melts almost immediately, allowing a portion of the steel to be drawn up into the evacuated space. Any dissolved gases and those produced by reactions between the components of the steel during freezing are liberated into the unfilled portion of the sample tube. These are subsequently removed through a suitable valve and analyzed. The sampling tube is so designed that a pressure considerably below atmospheric is produced after the gas evolution, resulting in a nearly complete removal of gas from the steel by the time solidification has occurred.

The sampling tubes tried out first in the development of the method consisted of steel tubes about 12 inches in length and 1½ inches inside diameter with a tubulation on the upper end closed with solder and kept cold during sampling by means of a wet cloth. A disk of copper or steel sheet was used to close the lower end. This tube was entirely too small to accommodate the gas evolved and its short length allowed steel droplets to reach and close off the exit, preventing removal of the gas for analysis. It was therefore necessary to modify the sampling apparatus to take care of these difficulties. The present sampling tube, although made up of several parts, is thought to be as simple as is compatible with successful sampling.

Construction of Sampling Tube

Fig. 1 illustrates the sampling apparatus now in use. The tubing used for Section A is first cut in 7-foot lengths and thoroughly cleaned. It was found necessary to use two sizes of tubing for this part, $\frac{11}{16}$ inch and ¾ inch respectively. The smaller tubing is used for tests of low FeO content and the larger for higher FeO content or colder steel. The clean tubes "A" are machined as follows: One end is machined on its inside diameter ⅛ inch deep to take a ⅞-inch copper disk cut from 0.005 to 0.007-inch thick copper sheet. The

other end is machined on its outside diameter to a diameter of 1 inch to take the trap member "B". About 4 inches below the same end, a ½-inch hole is drilled to receive the ½-inch copper tubing leading to the auxiliary reservoir "C". The copper disk is then silver soldered in place and the tube tested for leaks at this seal with compressed air.

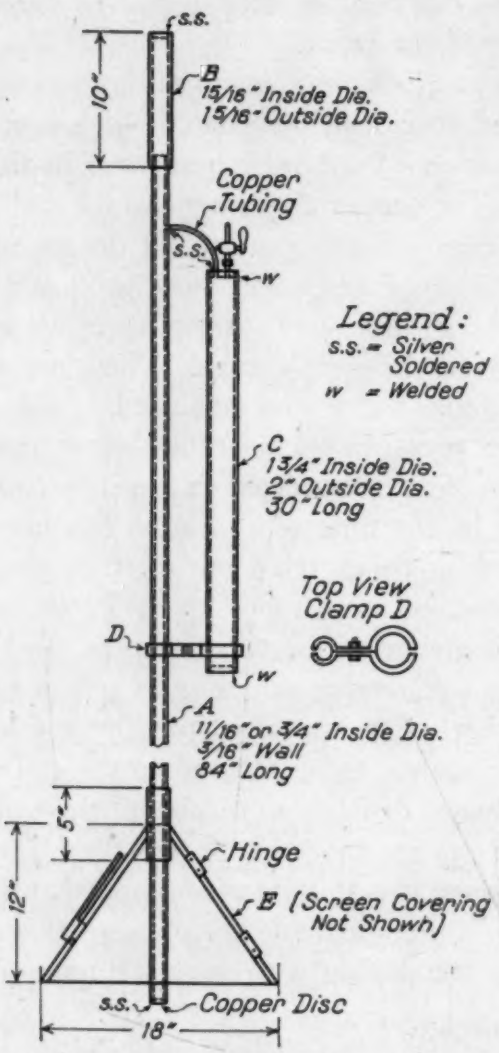


Fig. 1—Sketch of Sample Tube.

The trap member "B" is made from a 10-inch length of $\frac{5}{16}$-inch inside diameter steel tubing of $\frac{3}{16}$-inch wall, one end of which has been closed with a steel plug and silver soldered, the member being attached co-axially to the tube "A" by high melting silver solder. This trap member has two uses in this apparatus. It serves to entrap splashed steel during sampling and likewise allows tube "A" to be opened at both ends for cleaning.

Reservoir "C" at the side is made of a piece of steel or brass tubing, 2-inch outside diameter, 30 inches long, having ⅛-inch walls. Both ends of this tube are permanently closed by welded steel plates, the upper plate having two openings as shown, one to accommodate the copper tubing connection to the tube "A" and the other tapped with a ⅜-inch pipe tap to take the valve. This valve is an ordinary pressure stopcock with diagonal bore and is vacuum-tight when purchased but may require regrinding at intervals. The side reservoirs are all carefully made to size since their volume must be known as it enters into the calculation of the gas content. Their volume in our equipment is approximately 1400 cubic centimeters.

To assemble the parts, the copper connecting tube is silver soldered to the side reservoir "C" and the valve then soft soldered in place. The reservoir "C" is then clamped in proper position with respect to "A" and the copper tube silver soldered to "A", completing the assembly. The apparatus is then carefully tested for leaks by filling it with air at about 35 pounds pressure through the valve, which is then closed and the tube dipped in a large tank of water, any leaks being revealed by bubbles.

Safety Shield

There is no danger of splashing or spitting of steel during sampling if the tubes have been properly evacuated before use. However, if much air is present within the tube, the expansion when the molten steel is drawn in may blow the steel back out of the tube with considerable force. To effectively eliminate any such danger we have provided a detachable safety shield of heavy wire screen attached during sampling to the lower end of the sampling tube. This shield is shown in cross section in the diagram, being of conical shape in two halves hinged together. It is tightly held on tube "A" by means of the clamp shown at the left in Fig. 1.

Preparation of Tube for Sampling

The sampling tube is thoroughly evacuated through the valve using a Megavac pump and manometer. After evacuation it is removed from the pump after closing the valve and allowed to stand an hour or more, after which its vacuum is tested by connecting again to the evacuated manometer system and opening the valve. If no leak exists the tube is ready for use. A slight apparent leak may be

due to moisture within the tube, which must be removed by further evacuation. The entire tube is then weighed to an accuracy of 1 gram on a kilo beam balance of 20 kilograms capacity and its weight recorded against the valve number. The average weight of a sampling tube is about 11,000 grams. The length of tube "A" is also recorded, as it is needed for calculating the gas volume.

Side reservoirs and valves as well as trap members last indefinitely and may be used over and over. After each test, however, the tube "A", being partly filled with the steel sample which is welded to the lower end, but not elsewhere, must be cut shorter and the steel removed. It is then re-machined, a new copper disk soldered in, and reassembled for another test. About 3 to 4 inches of its length must be removed after each test. Our practice is to scrap the tube after six or seven tests, or when its length has been reduced to approximately 60 inches.

Sampling the Steel

An oversized open-hearth sampling spoon is ordinarily used. One with an 8-foot handle and holding about 8 or 9 pounds of steel after the spoon has been "slagged up" is suitable.

A sample of molten steel is taken from well toward the center of the bath and immediately after removal from the furnace any slag on the surface of the sample must be scraped away. The end of the sampling tube, which has been previously warmed by holding the lower end in the wicket hole of the furnace to assure the absence of any moisture, is then immersed an inch or two in the steel, but not touching the slag on the bottom of the spoon. When immersing the tube it should be held at an angle and slipped into the steel with a sidewise motion so as to avoid the possibility of an air bubble forming at the end of the tube. The copper disk melts almost immediately and a sample of the molten steel is drawn into the tube. After the steel is drawn in, the tube is moved in a circular motion in the spoon, describing an inverted cone, to cause a "blob" of steel to form and seal the end air-tight. This may require half a minute or more, after which the tube is removed from the spoon and the blob of steel hammered to assure a perfect seal. Before the steel is completely frozen it is well to throw a few chips of aluminum into the spoon to kill the remaining liquid steel and assure the formation of a nonporous seal on the tube.

Analytical Procedure

Figs. 2 and 2A illustrate the complete set of apparatus including the sampling and analytical equipment. In general the analytical apparatus consists of an all pyrex glass Toepler pump arrangement, shown in detail in Fig. 2A, for transferring the gases from the sample tube to the gas analysis system proper, shown on the right of

Fig. 2—Photograph of Gas Analysis Set-up Showing Position of Parts.

KEY

A—Stop-cock.
B—Stop-cock to Vacuum.
C—Sampling Tube in Position for Analysis.
D—Sampling Tube in Position for Evacuation.
E—Manometer for Recording Gas Pressure.
F—Manometer for Noting Vacuum.
G—Mercury Container—Lowered Position.
H—Shelf for Mercury Container—Raised Position.
I-I—Bulbs for Toepler Pump.
J—Burrell Gas Analysis Apparatus.
K—Megavac Pump.
L—Chart for Calculating Results.
M—Oxygen for Use in Gas Analysis.

Fig. 2. The Toepler pump system is of known volume and provided with a suitable manometer "E" for measuring the gas pressure and hence its total amount.

After the sample is taken the sampling tube is attached to the Toepler system by means of a rubber tube at stopcock "A", which is

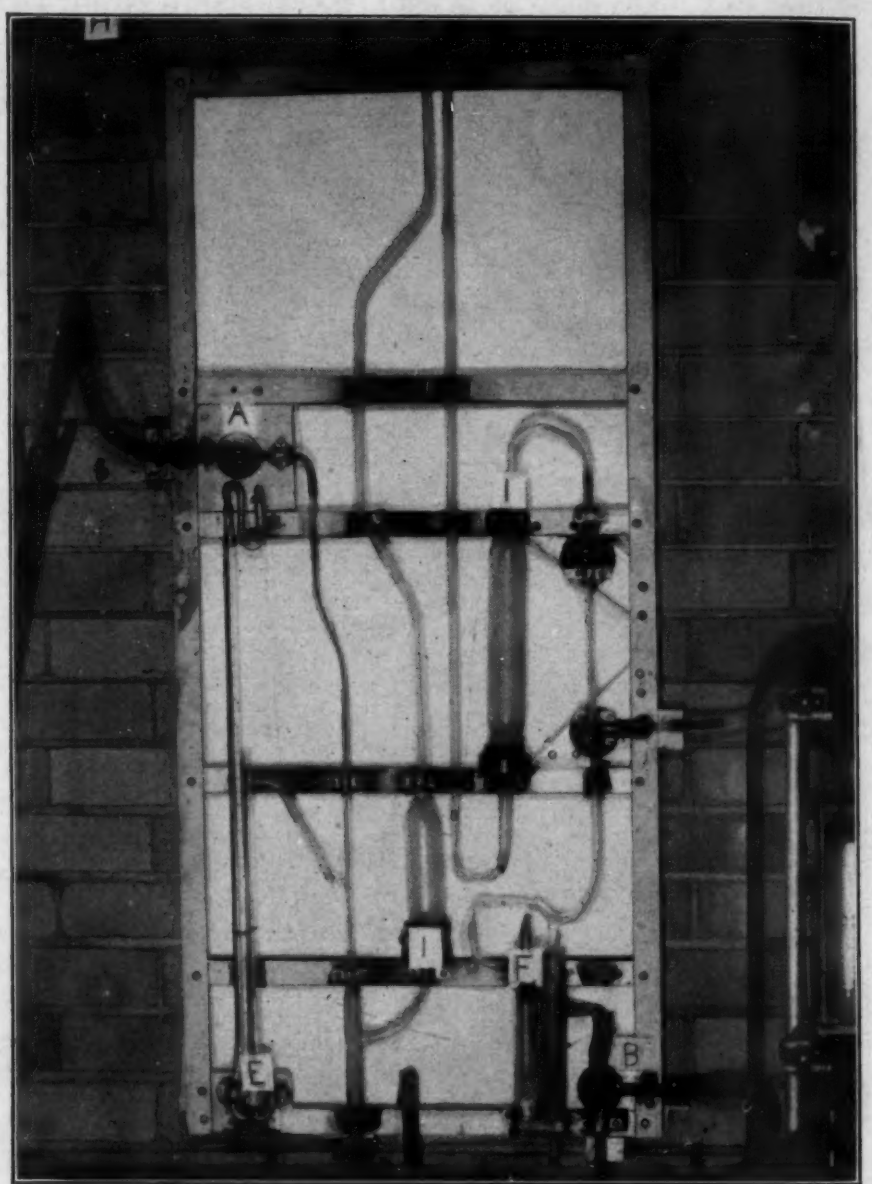


Fig. 2A—Close-up of Glass Toepler Pump Portion of Set-up Showing Construction.

opened and the system evacuated through stopcock "B" by the Megavac pump "K". The pump is then shut off from the system by closing the main stopcock "B" and the stopcock leading to the gas

Fig. 3—Photograph Showing Sampling of Steel for Gas Content.

analysis apparatus. The valve on the sampling tube reservoir is then opened, allowing the gas sample to expand into the Toepler system, where its pressure is recorded. By then raising and lowering the mercury in container "G" several times a portion of this gas is forced into the upper chamber "I" until about 100 cubic centimeters are collected. This is then forced into the Burrell gas analysis apparatus

"J" on the right and analyzed by standard procedures for CO_2, CO, H_2 and CH_4 successively as follows:

1. Pass through caustic soda solution.
2. Pass through ammoniacal $CuCl_2$ (two successive absorption bulbs).
3. Ignite in slow combustion pipette with known amount of oxygen.
4. Pass through concentrated H_2SO_4. (We frequently neglect this, calculating the loss of volume on ignition to H_2.)
5. Pass through caustic soda solution.

This procedure is not completely followed for routine analysis since both CO_2 and CH_4, being in small amounts, are neglected and only CO and H_2 are determined in steps 2 and 3. For research purposes, however, the analysis has frequently been made completely.

It was originally the procedure to absorb any oxygen that might be present by means of pyrogallic acid solution as the first step in the gas analysis and to determine the nitrogen by difference at the end of the analysis. Oxygen, however, is never present unless due to small leaks of air into the apparatus and the amount of nitrogen is usually very small. Further, if air leaks into the apparatus while the steel is hot the oxygen will be completely burned by the hydrogen present without apparently affecting the CO content thus leaving the nitrogen in the sample. We have not been able to prove, therefore, whether the small amounts of nitrogen present, which are seldom over 1 per cent of the total gas volume, are actually evolved from the steel or not.

The so-called CH_4 present never runs over 1 or 2 per cent of the total gas volume and may consist partly of acetylene, etc. Frequently the odor of arsine or similar gases may be detected in the sample, but analysis does not show them to be present in appreciable quantities. The CO_2 present is usually 2 per cent or less.

Calculation of Results

The results of gas analysis are calculated in terms of per cent by weight of the steel sample. For this purpose the following factors are required:

1. *Volume of entire apparatus when total pressure is recorded.* This is the sum of several volumes as follows:

(a) Volume of auxiliary gas reservoir "B".

(b) Volume of tube "A" less volume of steel sample present. The volume of tube "A" is calculated from its measured length and

its known inside diameter. The volume of the steel sample is calculated from its weight and specific gravity.

(c) Volume of entire Toepler pump system.

2. *Pressure of gas at the above volume.* This pressure is recorded directly on the manometer "E".

3. *Weight of the Steel Sample.* This is obtained by weighing the entire sampling tube before and after the test. The difference in weight must, however, be corrected for the weight of the blob of steel frozen on the bottom which presumably did not contribute to the gas volume. The sample apparatus is therefore hung on a hook and the volume of the blob of steel estimated by immersing it in water in a graduated litre cylinder to a depth of 4 inches and noting the volume displaced. This volume displacement must be corrected for the normal displacement of 4 inches of the tube itself.

4. *The percentage analysis (by volume) of the gas mixture.* This is obtained by the standard method described above.

While the description of the calculation appears complex, the actual time and trouble of calculating is largely eliminated in practice by the use of a nomograph as shown in Fig. 4. This chart will be seen to be composed of four graphs, properly coordinated, and may be used for calculations as follows:

1. The total volume of the gas when the pressure is recorded is calculated by adding the following items:

(a) Volume of Toepler pump system
(b) Volume of sampling tube side reservoir
(c) Length of tube "A" times volume per unit length
(d) Volume of small trap reservoir

From the total of these volumes

(e) Subtract ⅛ of the steel sample, corrected for blob.

The result is recorded as Total Corrected Volume.

This volume has been graphed as a series of diagonal lines in the lower left quadrant of the nomograph (#1). Along the upper coordinate of this quadrant is plotted "Inches of Pressure" as read on the manometer. By reading vertically downward from "Inches of Pressure" to the proper volume diagonal, thence horizontally to the central vertical axis the true volume of the gas sample at atmospheric pressure is obtained.

2. The volume of the desired constituent (e.g., CO) is then read from the lower right quadrant (#2). Radial lines refer to per cent by volume of the desired constituent as obtained from the gas

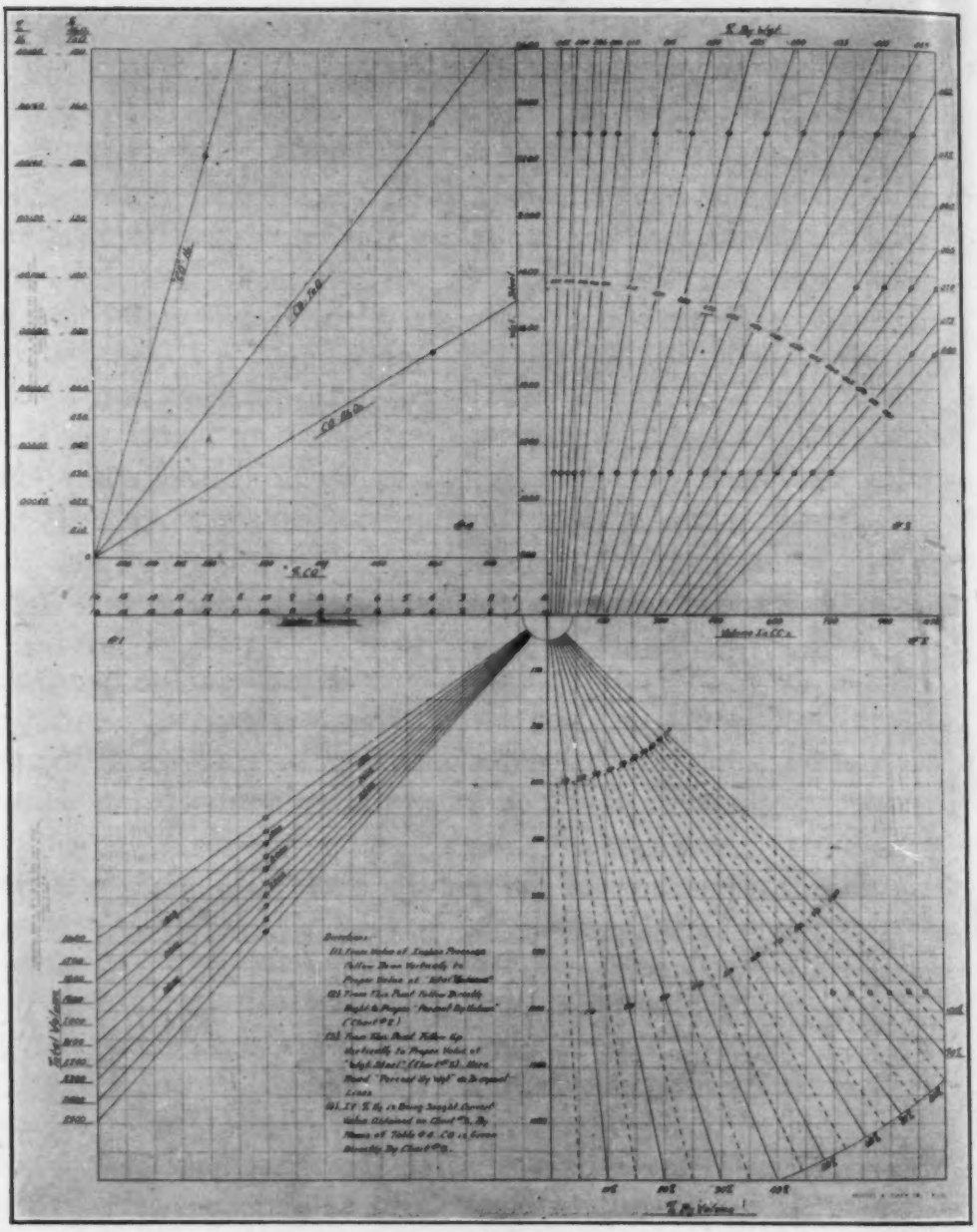


Fig. 4—Nomograph for Rapid Calculation of Gas Content Data.

analysis. By reading horizontally from the total volume previously read to the proper per cent diagonal, thence vertically upward to the horizontal axis, the volume of the desired constituent is given.

3. The per cent by weight in steel of the desired constituent is read from the upper right quadrant (#3). Here the radial lines refer to gas per cent by weight in steel through the range 0.000 to 0.080 per cent. The vertical axis shows the "weight of steel" sample, obtained as described above. Hence, by reading vertically upward

from volume of constituent (horizontal axis) to the proper value of "weight of steel," the "per cent by weight" is obtained by interpolation between the radial lines.

4. The value thus given for "per cent by weight" is numerically correct for either per cent CO or per cent N_2, but in the case of hydrogen must be multiplied by a factor of molecular weight by means of the upper left quadrant (#4). Here the value of "per cent by weight" from the upper right quadrant is plotted horizontally as "per cent CO." By reading vertically from the horizontal axis to the line "CO — H_2," and thence horizontally to the vertical axis, the per cent H_2 is read directly.

On this same quadrant the values of per cent CO may be calculated to per cent FeO, or for purposes of comparison with the results of the Bureau of Mines Aluminum Method for FeO in Steel,[5] may be calculated to per cent Al_2O_3.

The whole calculation may be done by this graphical method in a minute or two. In fact, we have found that the entire analysis of gas in molten steel from the time of taking the sample until the results are completely calculated may be done, if necessary, in approximately twelve minutes.

Results

The results of our work on steels having a carbon content of 0.15 per cent or over clearly indicate that under the conditions of test all but a small fraction of the FeO present is evolved as CO gas when the steel freezes. Hence by multiplying the per cent by weight of CO by a factor of 2.55, which is the ratio of molecular weights, a value for the per cent of FeO may be obtained as accurately as by any method now available. This has been checked by analyzing simultaneous samples by the Bureau of Mines Aluminum Method. As illustrated in the following table, the checks are in most cases very satisfactory. The gas method described in this paper is found to give somewhat lower results after aluminum deoxidation than the Bureau of Mines Method, which is to be expected as the Al_2O_3 inclusions from deoxidation contaminate the test. In such a case the results of the gas method would be more dependable.

The accurate check which may be obtained between FeO values run by the gas method and the Bureau of Mines method may be noted in the following set of tests taken alternately from a heat of 0.65 per cent carbon open-hearth steel.

[5] C. H. Herty et al, Bureau of Mines, Report No. 3166, 1932.

Table I
Analysis of Gas Evolved

Time	Per Cent Carbon in Bath	Per Cent CO	Per Cent H_2	Calc. Per Cent FeO	Per Cent FeO by Bur. of Mines Method
8:20	1.04	.....	.....	.....	0.118
8:23	.....	0.042	0.00037	0.108	.....
8:30	1.01	.....	.....	.....	0.178
8:33	.....	0.073	0.00083	0.186	.....
9:20	0.78	.....	.....	.....	0.178
9:22	.....	0.068	0.00180	0.168	.....
9:30	0.725	.....	.....	.....	0.160
9:32	.....	0.063	0.00050	0.161	.....
9:40	0.685	.....	.....	.....	0.178
9:42	.....	0.067	0.00035	0.173	.....
After deoxidation, tests before tap showed—					
11:15	0.65	.....	.....	.....	0.079
11:17	.....	0.029	0.00080	0.074	.....

It should be emphasized that we have not always been able to obtain checks by the two methods as accurately as in the case of the heat shown, a fact which we have had to ascribe to lack of care in taking the tests by one method or the other or to errors in analysis of the Al_2O_3 by the Bureau of Mines Method.

The values for FeO increased to a steady value after about 1.00 per cent carbon was reached, though the carbon continued to drop. The usual drop in FeO during furnace deoxidation is indicated by the last test. Hydrogen values were rather erratic, as is often the case, showing a period of high content during a portion of the working of the heat. The increase in hydrogen after deoxidation is typical, although rather low in this heat as compared to others. In general, during a heavy boil the hydrogen is fairly low (around 0.0005 per cent or less) and frequently rises well above 0.001 per cent after the heat has been killed.

The variation of gas content in two types of electric furnace steel is illustrated by the examples shown in Table II. A high carbon-chromium steel and a low carbon-nickel-molybdenum steel are shown.

Table II

Test	Time	Analysis of Evolved Gases Per Cent CO	Per Cent H_2	Per Cent FeO Calc. from CO
	Heat 11420 1.00 C. 1.75 Cr.			
1	Before slag-off	0.037	0.00050	0.095
2	1 hour under carbide reducing slag	0.015	0.00080	0.039
3	2 hours under reducing slag	0.011	0.00070	0.027
4	At tap	0.010	0.00100	0.025
	Deoxidizers were added in the ladle.			
	Heat 37932 4615 Specification			
1	Before slag-off	0.034	0.00027	0.087
2	Before silicon deoxidation	0.023	0.00114	0.059
3	Before aluminum deoxidation	0.009	0.00135	0.022

The decrease in FeO under the carbide slag and the simultaneous increase in hydrogen are clearly shown in both heats.

Conclusion

The method of gas analysis described in this paper has proved very useful in determining the degree of deoxidation in electric furnace and open-hearth steel and the effect of various deoxidation practices and slags on the amount of FeO present in the steel at tap. It is also of considerable value as a tool in working out the best furnace practice for a given specification of steel. For example, it has been found, as might be surmised, that the type of inclusion in steel is a direct function of the per cent of FeO present at the time of final deoxidation. We feel that this method of gas analysis of molten steel is suitable for production control as well as for use in research.

CASE HARDENING BY DIRECT TRANSFORMATION OF AUSTENITE

By Bernard R. Queneau and Ralph L. Dowdell

Abstract

This paper deals with a practical phase of case hardening by quenching special steels into a bath of tempering salt maintained at temperatures between 600 and 400 degrees Fahr. depending on the hardness desired. With this method stronger and tougher case hardened steels will result with hardness in the neighborhood of 60 Rockwell C. Various figures and tables describe the results in considerable detail.

Introduction

THE main purpose of carburizing steel is to obtain, through the penetration of carbon, an outside portion high in carbon, generally known as the case, which gradually decreases in carbon content towards the center of the piece. After suitable heat treatment, this case is hard and resistant to wear. Most literature on the subject recommends that the carbon content of the core be kept below 0.20 per cent yet few reasons are given for this low value. Higher carbon contents in the core produce more brittleness because of the increased stresses between the case and the core obtained by quenching.

Woodvine[1] actually showed that even with a low carbon core the strength of the specimen is increased when the core is removed. To increase the strength of the low carbon core, it has been necessary to add different alloying elements such as chromium, nickel, vanadium, and molybdenum, all of which increase the cost of the steel. Instead of using expensive alloying elements the strength of the core could be increased by increasing the carbon content if the specimen could be heat treated without introducing stresses which occur upon quenching

[1]J. B. R. Woodvine, "The Behavior of Case Hardened Parts Under Fatigue Stresses," Iron and Steel Institute, Carnegie Scholarship Memoirs. Vol. XIII, 1924.

This paper is the summary of a Ph.D. thesis by Bernard R. Queneau, graduate student of the University of Minnesota, and Ralph L. Dowdell, professor of Metallography, University of Minnesota, Minneapolis, Minn. Manuscript received June 24, 1936.

into a medium at room temperature. The transformation of austenite at some elevated temperature has been shown by Davenport and Bain[2] to produce a structure that has high strength and great toughness.

The purpose of this investigation was to attempt to transform the austenite in the case of carburized steel by quenching into a bath at the proper subcritical temperature and holding until the transformation was completed. A study of the physical properties obtained and a comparison of these properties with those of quenched and tempered case carburized steels was made.

Experimental Procedure and Results

Steels Used

Four steels of increasing alloy content were used, a plain carbon steel of 0.64 per cent carbon, a carbon steel of 0.42 per cent carbon containing 0.20 per cent molybdenum, an S.A.E. 2330, and an S.A.E. 3120 steel. Their composition, form and key letter are given in Table I.

Table I
Composition of Steels

Steel	Type	C	Mn	P	S	Si	Ni	Cr	Mo	Form, Diameter of Rounds Inches
A	Carbon	0.64	0.80	0.017	0.030	0.15	....	....	....	1
B	Carbon-Molybdenum	0.42	0.77	0.020	0.030	0.20	....	....	0.20	1 1/8
C	S.A.E. 2330	0.32	0.65	0.007	0.021	0.16	3.45	....	....	3/4
D	S.A.E. 3120	0.18	0.65	0.011	0.024	0.16	1.20	0.64	....	3/4

As the manganese content of the four steels was approximately the same and the phosphorus, sulphur and silicon contents low, the reaction rates of the steels were not affected by them to any large extent. It should be noted that the plain carbon steel had a carbon content much higher than that used in general practice; the molybdenum steel also was high in carbon with only the S.A.E. 3120 having a composition usual in case hardening steels.

[2]E. S. Davenport and E. C. Bain, "The Transformation of Austenite at Constant Subcritical Temperatures," *Transactions*, American Institute of Mining and Metallurgical Engineers, Vol. 90, Iron and Steel Division, p. 117-154.

Table II

Grain Size and Normalities of the Steels

Steel	A.S.T.M. Grain Size	Normality
A	3	Normal
B	<8	Normal
C	4–<8	Large grains normal Small grains abnormal
D	1–7	Normal

McQuaid-Ehn tests were made on the four steels and results obtained are given in Table II, and Figs. 1 to 4 show the cases at 1000 diameters.

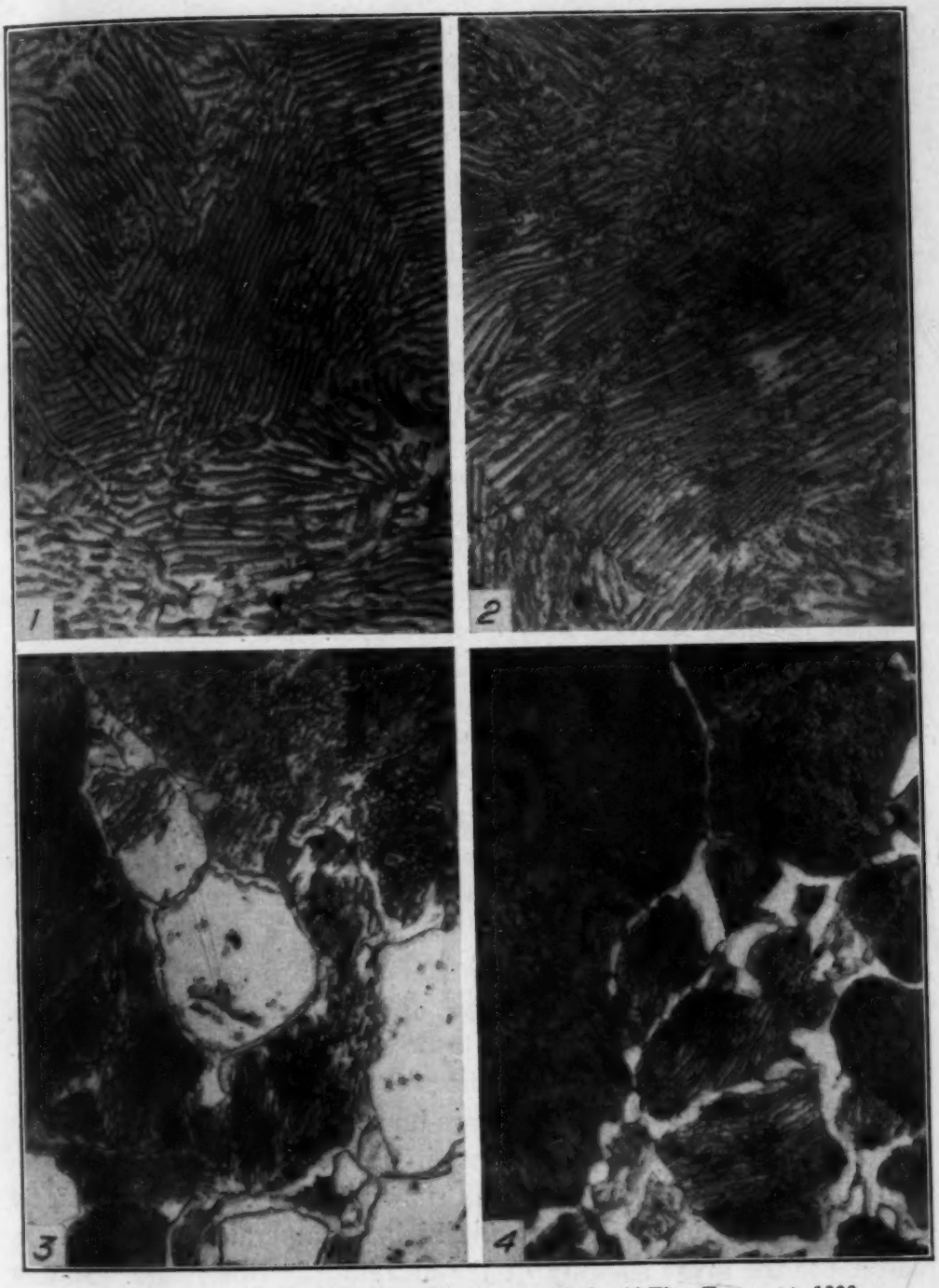
The plain carbon steel had a large grain size, the molybdenum steel was *peculiar* in the fact that no hypereutectoid case was formed during the 8 hours carburizing at 1700 degrees Fahr. The nickel steel was *peculiar* in that the large grains were normal while the small grains were completely abnormal. The nickel-chromium steel also showed a duplex structure but all the grains were normal.

Reaction Rates of the Steels

To know the time required for the transformation of the austenite, it was necessary to determine the reaction rates of the four steels. A knowledge of the reaction rates was also useful in the study of the hardenability of the steels. The method used in determining the reaction rates of the steels was the one developed by Bain and Davenport. To determine the rate of transformation of the austenite, small specimens were necessary so that they could acquire very rapidly the temperature of the bath into which they are quenched.

The 1-inch and $1\frac{3}{16}$-inch rounds of steels A and B were forged to ½-inch square bars and hot-rolled to 0.150-inch squares. The ¾-inch rounds of steels C and D were hot-rolled directly to 0.150-inch squares. The bars were then rolled to flats of 0.100-inch thickness and cut to 1½-inch lengths. The specimens were then normalized by holding one-half hour at 1700 degrees Fahr. and cooled in a small carburizing box in order to eliminate as much as possible the effects of forging and rolling. To eliminate all surface defects introduced in the working, the specimens were surface ground 0.010-inch on both sides, and were then numbered for identification and notched.

As we were interested in determining the reaction rate of the



Photomicrographs Showing Normality After McQuaid-Ehn Test. × 1000.
Fig. 1—Steel A. Plain Carbon.
Fig. 2—Steel B. Carbon-Molybdenum.
Fig. 3—Steel C. S.A.E. 2330.
Fig. 4—Steel D. S.A.E. 3120.

case only, all the small specimens were carburized 8 hours at 1700 degrees Fahr. and cooled rapidly to prevent the formation of carbide

network. The carbon contents of these specimens were on the whole higher than the greater part of the case because the specimens were hypereutectoid even at the center after carburizing.

These specimens were then reheated individually to 1700 degrees Fahr. in a carburizing box (to prevent scale) and quenched directly into the lead pot with its temperature closely controlled. To investigate the rate of transformation at the lower temperatures, a lead-tin-bismuth alloy was used for the quenching medium. The progress of the transformation was determined entirely by use of the microscope though some difficulty was encountered at the lowest temperature, 400 degrees Fahr., determining when the transformation was completed. Figs. 5 to 8 give the reaction rates of the four steels as carburized.

A few typical microstructures are shown in Figs. 9 to 14, all of which are of partly transformed specimens showing the structure in more detail.

The dark acicular needles shown in Fig. 9 are typical of the product obtained at the lower temperatures. Fig. 10 shows clearly the two modes of transformation. This specimen was not quenched with sufficient rapidity to miss the upper transformation range entirely and thus some of the austenite transformed to very fine pearlite, shown as the nodular constituent. The austenite that was retained to the lower temperature started to transform after a considerable passage of time to the acicular constituent. The remaining austenite upon quenching transformed to martensite, the white background. The structures at 800 degrees Fahr. are frequently formed of partly nodular and partly acicular constituents because of the difficulty of obtaining a successful quench at just below the nose of the S-curve. Fig. 11 shows the austenite transformed to the two structures. Fig. 12 shows the transformation of austenite to fine nodular pearlite, the transformation beginning at the grain boundary.

The nickel-chromium steel precipitated carbide from the austenite at 1000 degrees Fahr. or higher as shown in Figs. 13 and 14. The precipitation of this carbide at the higher temperatures might be a reason for the great stability of the austenite in this range. It was not in the scope of this investigation to determine the effect of the carbide precipitation but the point is a very interesting one.

It is interesting to compare the rate of reaction of the four steels from the point of view of alloy content. If we select the plain carbon steel as a standard, we see that the 0.20 per cent molybdenum steel does not affect the rate to any large extent in the steels as carburized.

The addition of 3.5 per cent nickel delayed the beginning of transformation from 3 seconds to 8 seconds at 1000 degrees Fahr., but at 400 degrees Fahr. the transformation was complete for all three steels in approximately 5 hours. Thus the presence of nickel in these high carbon steels had only a minor effect on the rate of transforma-

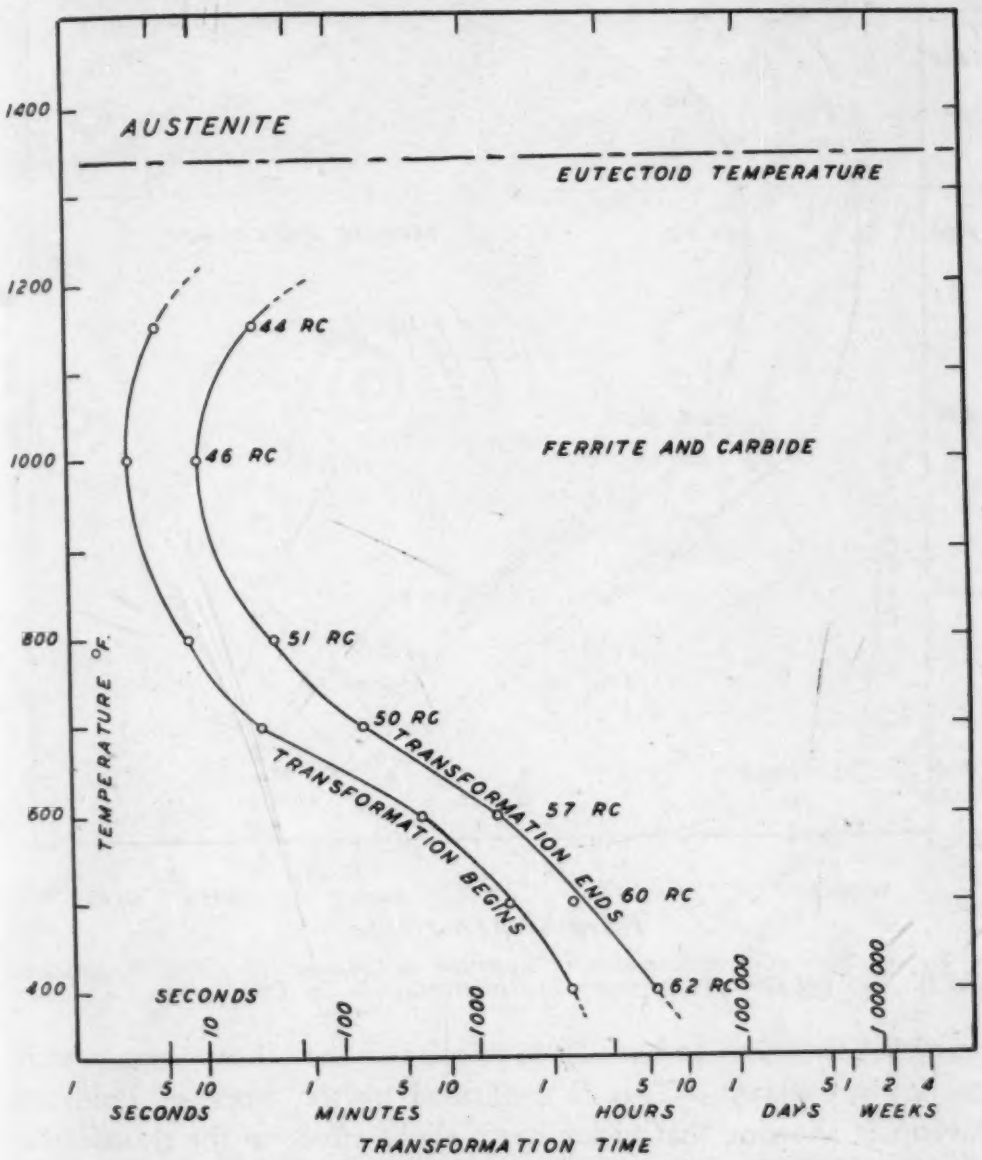


Fig. 5—Time of Transformation of Austenite at Constant Subcritical Temperatures. Steel A. Plain Carbon—As Carburized, 1.42 Per Cent Carbon.

tion. The nickel-chromium steel, however, has a considerably slower rate of reaction requiring 48 hours for completion at 400 degrees Fahr. and 40 seconds at 900 degrees Fahr. The 0.64 per cent chromium present is the main factor in retarding the rate of reaction since

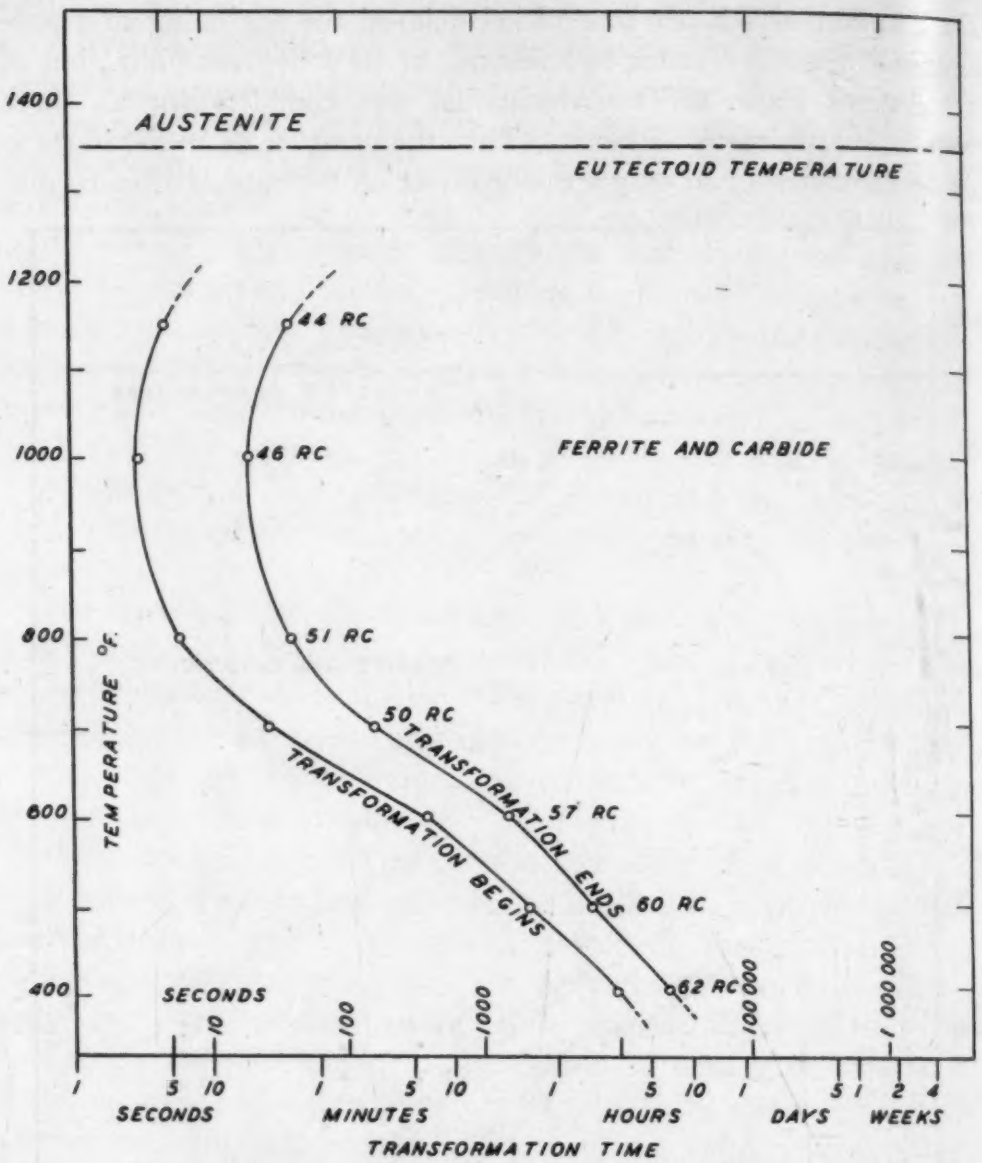


Fig. 6—Time of Transformation of Austenite at Constant Subcritical Temperatures. Steel B. 0.20 Per Cent Molybdenum—As Carburized, 1.37 Per Cent Carbon.

the nickel was seen to have little effect and the other elements were practically constant. This is confirmed by the work of Bain and Davenport showing that nickel has a slight effect on the transformation rate while chromium has a very large effect.

Hardenability

To determine whether the steels with large cross section would harden by the use of an interrupted quench, standard 1-inch rounds were used. The ¾-inch rounds of steels C and D were upset to 1

inch and normalized at 1700 degrees Fahr. after one-half hour. The specimens were machined to size and were 3 inches long.

A notch of 0.075 inch was cut in the middle to facilitate breaking of the specimens. Two samples of each steel were carburized 8 hours at 1700 degrees Fahr. and heat treated as follows: Steels

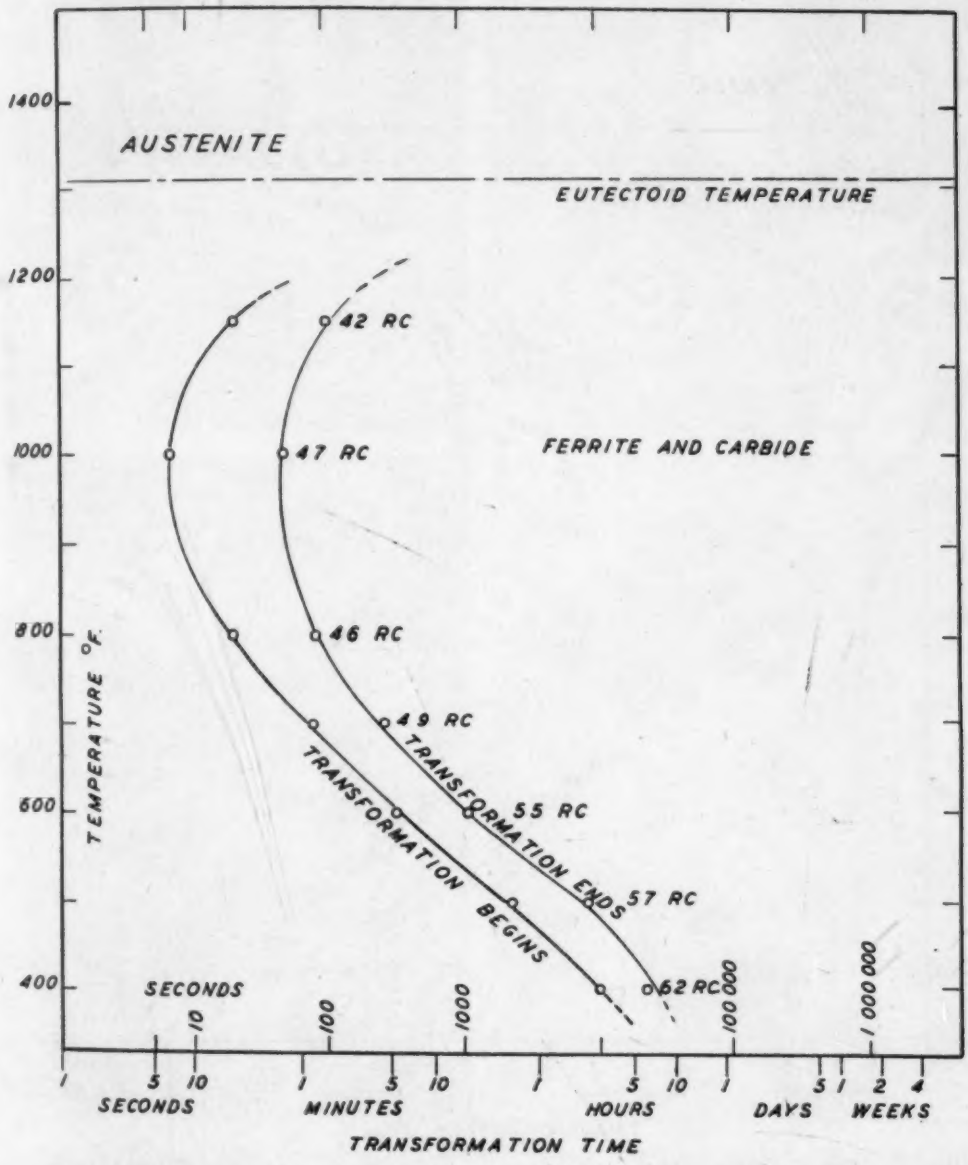


Fig. 7—Time of Transformation of Austenite at Constant Subcritical Temperatures. Steel C. 3.45 Per Cent Nickel—As Carburized, 1.47 Per Cent Carbon.

A and B were quenched in water and C and D were quenched in oil. Other specimens of steels A, B and C were quenched in a bath of tempering salt (sodium and potassium nitrate) and held 5 hours at

400 degrees Fahr. Steel D was quenched into the salt bath and held 48 hours at 400 degrees Fahr.

The steels were then broken, polished and a study made of the Rockwell "C" hardness across the section. The results of these readings are shown in Figs. 15 to 18. Steels A and D did not harden

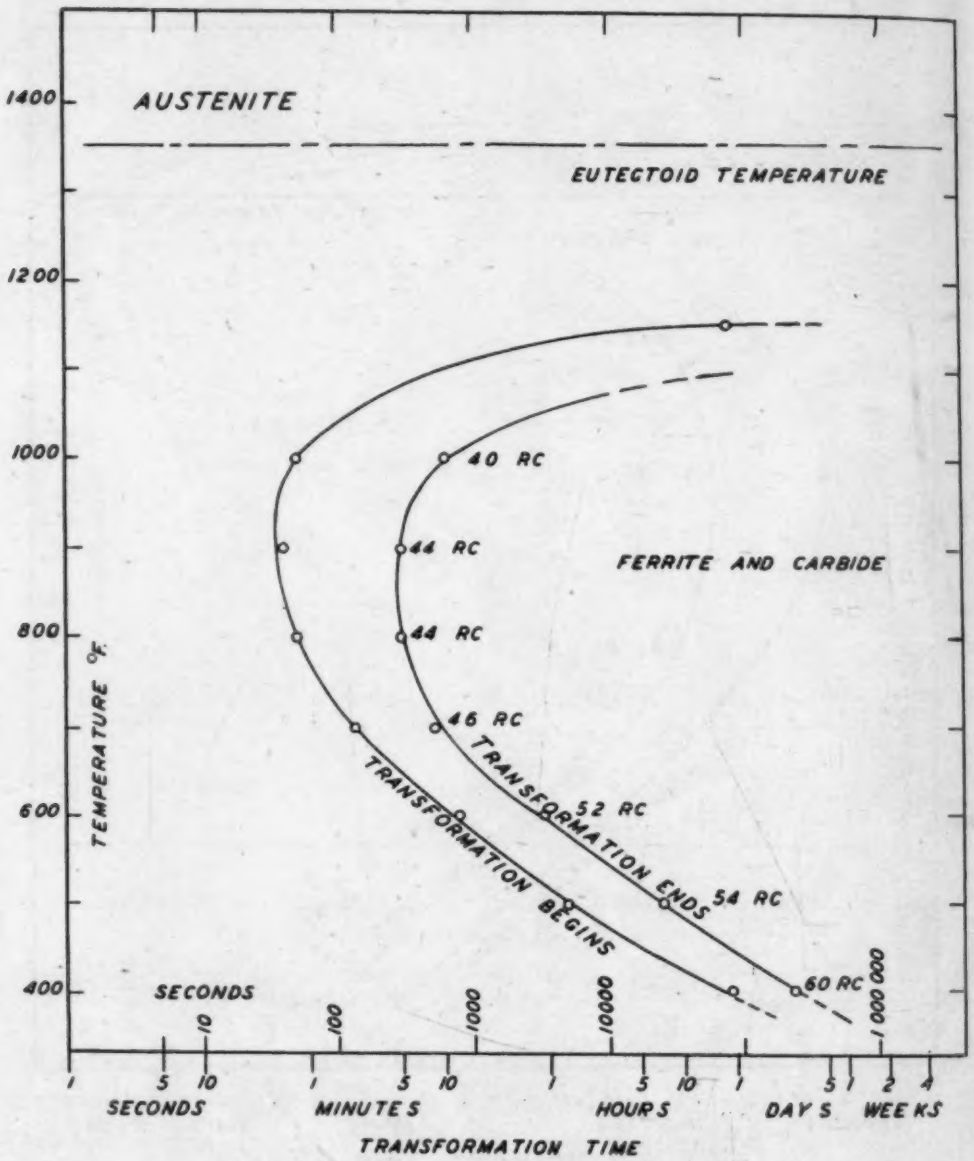


Fig. 8—Time of Transformation of Austenite at Constant Subcritical Temperatures. Steel D. 1.20 Per Cent Nickel, 0.64 Per Cent Chromium—As Carburized, 1.02 Per Cent Carbon.

at all by the interrupted quench method. Steel C hardened at the surface, but the hardness dropped off very rapidly. Steel D, however, developed a hardenability curve very similar to that of the

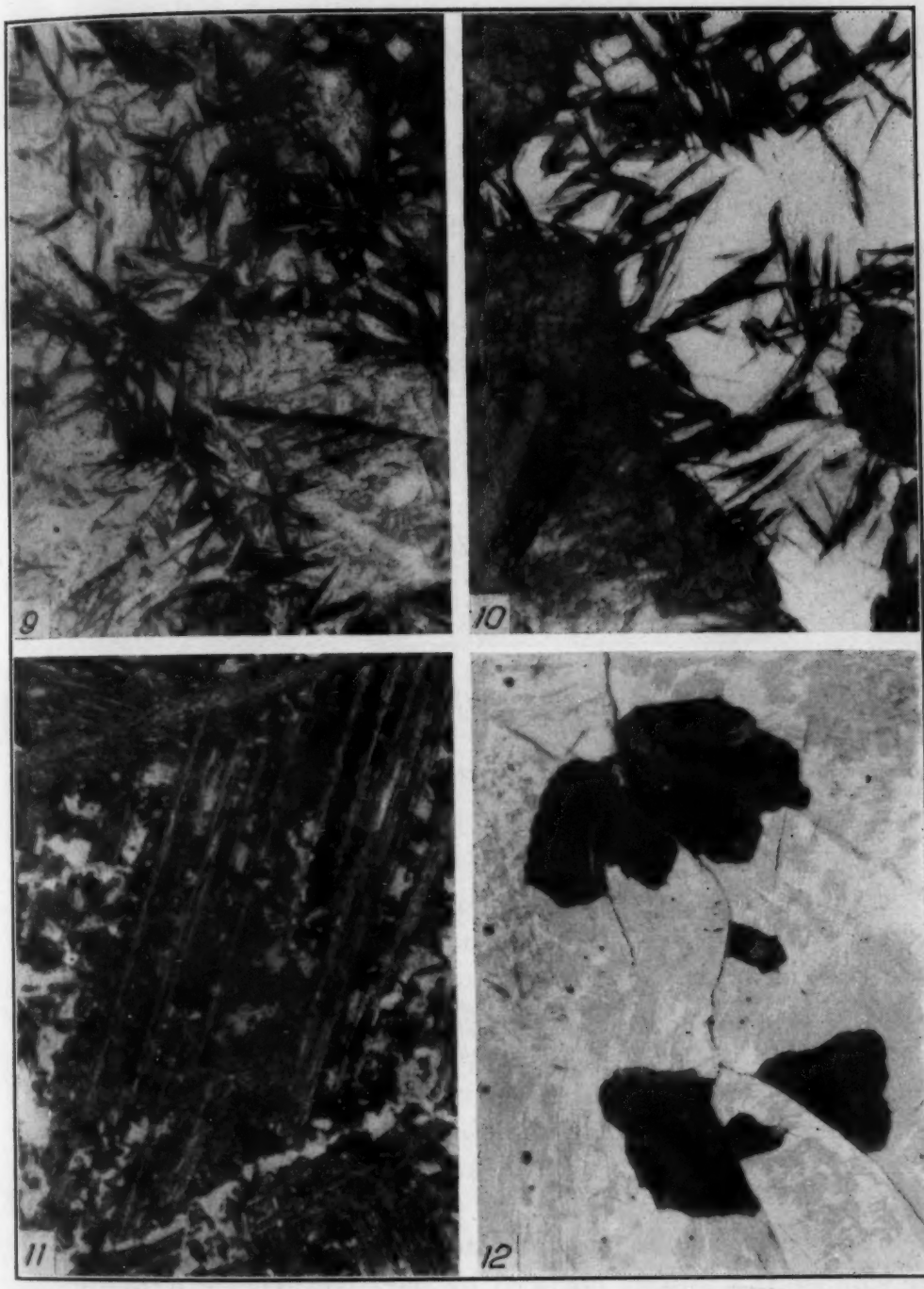


Fig. 9—Steel A. Plain Carbon. Carburized to 1.42 Per Cent Carbon. Quenched from 1700 Degrees Fahr. into Lead Alloy Bath at 400 Degrees Fahr. and Held 14,400 Seconds, and Finally Quenched in 5 Per Cent NaOH. × 1000.

Fig. 10—Steel A. Plain Carbon. Carburized to 1.42 Per Cent Carbon. Quenched from 1700 Degrees Fahr. into Lead Alloy Bath at 500 Degrees Fahr. and Held 2000 Seconds, and Finally Quenched in 5 Per Cent NaOH. × 1000. Speed of Quenching Insufficient to Prevent Formation of Nodular Pearlite.

Fig. 11—Steel C. 3.45 Per Cent Nickel. Carburized to 1.47 Per Cent Carbon. Quenched from 1700 Degrees Fahr. into Lead Alloy Bath at 800 Degrees Fahr. and Held 70 Seconds, Finally Quenched in 5 Per Cent NaOH. × 1000.

Fig. 12—Steel C. 3.45 Per Cent Nickel. Carburized to 1.47 Per Cent Carbon. Quenched from 1700 Degrees Fahr. into Lead Alloy Bath at 1150 Degrees Fahr. and Held 25 Seconds, Finally Quenched in 5 Per Cent NaOH. × 1000.

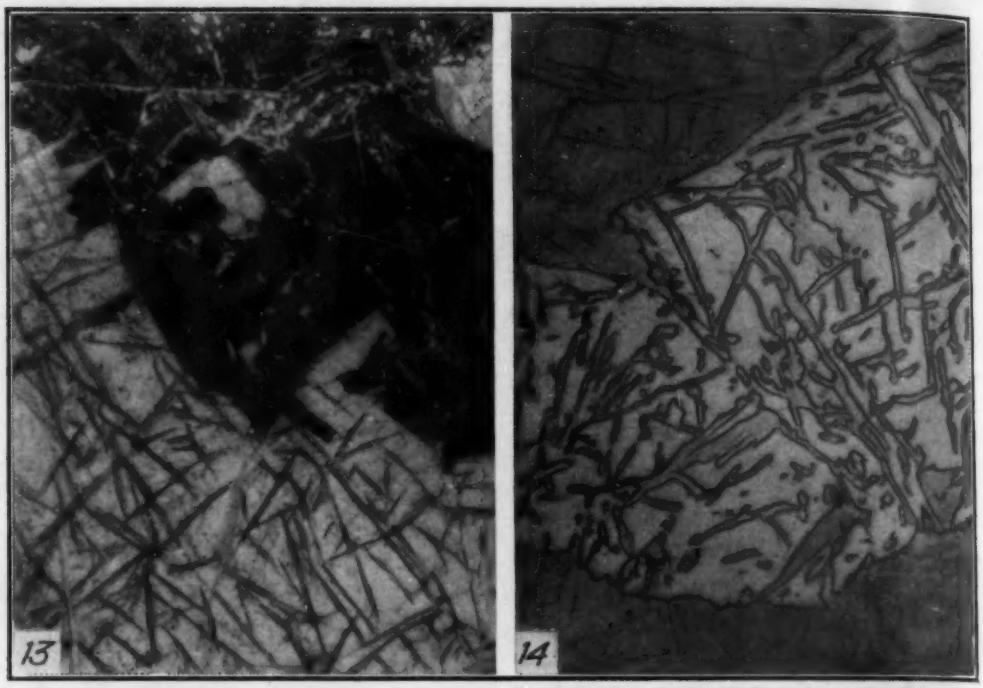


Fig. 13—Steel D. 1.20 Per Cent Nickel, 0.64 Per Cent Chromium. Carburized to 1.02 Per Cent Carbon. Quenched from 1700 Degrees Fahr. into Lead Alloy Bath at 1000 Degrees Fahr. and Held 240 Seconds, Finally Quenched in 5 Per Cent NaOH. × 1000.
Fig. 14—Steel D. 1.20 Per Cent Nickel, 0.64 Per Cent Chromium. Carburized to 1.02 Per Cent Carbon. Quenched from 1700 Degrees Fahr. into Lead Alloy Bath at 1150 Degrees Fahr. and Held 92,000 Seconds, Finally Quenched in 5 Per Cent NaOH. × 1000.

quenched and tempered specimen, showing just a little more softness in the center.

Since steels A and B did not harden it was decided to try a smaller size specimen. A ¾-inch round was used and an identical heat treatment was given. In spite of the reduced cross section, steels A and B again did not harden by the interrupted quench method. The nickel steel, however, hardened in the case completely and in fact at the surface was a little harder than by quenching and tempering due to the retention of austenite by the latter method. The nickel-chromium steel D was again very similar by both methods of heat treatment.

By a study of the rates of reaction of the four carburized steels we can see that results obtained by the hardenability tests are consistent. The carbon steel and the carbon-molybdenum steel start to transform within 5 seconds over the 300-degree temperature range of 1150 to 850 degrees Fahr. This time is insufficient, therefore, to lower the temperature of the case of the steels in a salt bath at 400 degrees Fahr. before transformation takes place in either 1-inch or ¾-inch rounds. The nickel steel, however, starts to transform

only after 10 seconds in a 200-degree Fahr. temperature range. The quenching is not drastic enough to obtain a case that does not transform except at the very outer portion. In the ¾-inch round, however, the case temperature was lowered to 400 degrees Fahr. without any transformation taking place. A study of the nickel-chromium steel shows that there is considerable time before transformation begins, so that even a larger section than 1 inch could in all probability be hardened by either method.

Charpy Impact

For the impact tests both notched and unnotched specimens were used in an effort to obtain, if possible, the impact values of the case. Due to the great depth of the notch on the standard Charpy specimen, a special rounded notch with a depth of only 0.079 inch was used. This notch was made by placing the specimen on a surface grinder and using, instead of the usual stone, a thin cutting-off wheel. The notches were cut on all the specimens of the same steel at the same time and were, therefore, quite uniform. To test whether this notch was satisfactory, four trial specimens were made of steel A, heat treated and broken. The results were within one degree of each other. The specimens were made by forging the rounds to ½-inch squares and surface grinding to 0.394-inch squares

Table III
Charpy Impact Tests on Case Hardened Specimens

Carburized at 1700 degrees Fahr.—8 hours and treated as shown

Steel	Heat Treatment Quenched in	Impact Resistance in Foot-Pounds Notched	Unnotched	Depth of Case Inches	Rockwell "C" Hardness of Case
A					
Plain Carbon	Water[1]	11	7	0.055	64
0.64 Per Cent Carbon	Salt[2]	9	10	0.055	62
B					
C-Mo Steel	Water[1]	7	12	0.042	65
0.42 Per Cent Carbon	Salt[2]	15	11	0.042	62
0.20 Per Cent Molybdenum					
C					
S.A.E. 2330	Oil[1]	8	25	0.035	60
	Salt[2]	8	26	0.035	62
D					
S.A.E. 3120	Oil[1]	8	13	0.055	63
	Salt[2]	7	10	0.055	62

[1]All samples quenched from the box at 1700 degrees Fahr. and those quenched in oil or in water were tempered 1 hour at 325 degrees Fahr.

[2]The salt bath was held at 400 degrees Fahr. and samples of steels A, B and C were held 5 hours while samples of steel D were held 48 hours.

after normalizing as usual. Two notched and two unnotched specimens were made for both heat treatments of each steel. Results of Charpy tests are given in Table III.

The most striking point that was first noticed was the uniformity of results irrespective of steel used, method of heat treat-

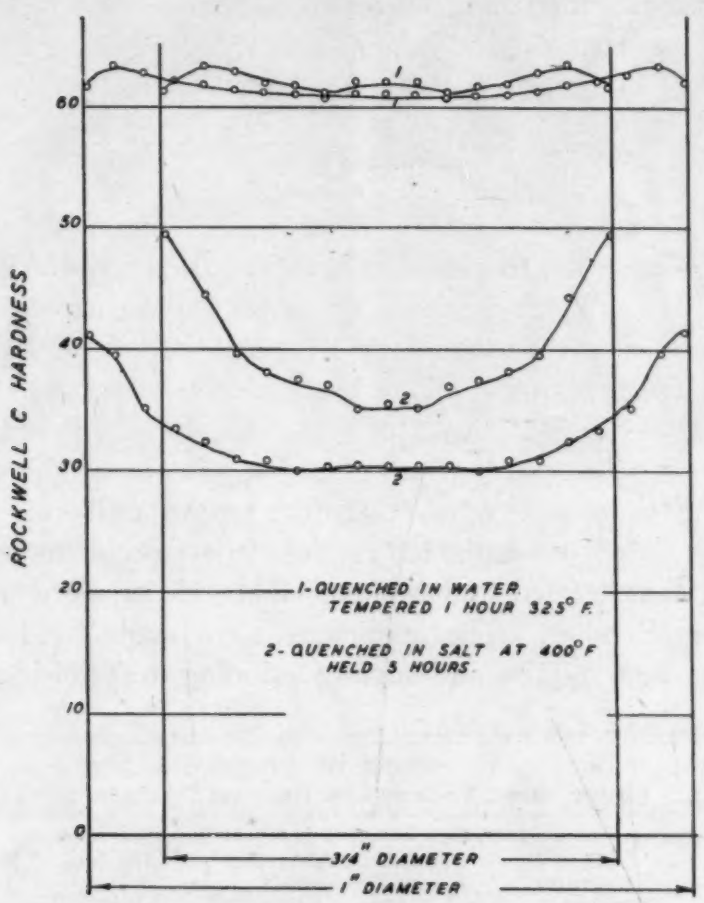


Fig. 15—Steel A. 0.64 Per Cent Carbon Steel Carburized 8 Hours at 1700 Degrees Fahr.

ment and whether notched or unnotched. The unnotched nickel steel gave considerably higher results for the only real exception, though there was a slight increase in the nickel-chromium steel. This checks with work done by McMullan[3] which also showed a great sensitiveness of the nickel steels to notching. It appears that the values obtained for the notched steels gave results that would tend to show that in these hardened steels the impact values were approximately the same irrespective of alloy content as obtained by

[3]O. W. McMullan, "Physical Properties of Case Hardened Steels," TRANSACTIONS, American Society for Metals, Vol. 23, 1935, p. 319.

the Charpy test. This was also checked by Birchfield[4] when he obtained for three steels, namely S.A.E. 2315, 2512 and 4615, impact values of 8, 10 and 10 foot-pounds. For the S.A.E. 4820 he obtained a value of 14 foot-pounds (all Izod values) and decided that there was a different value for each steel. Due to the delicacy

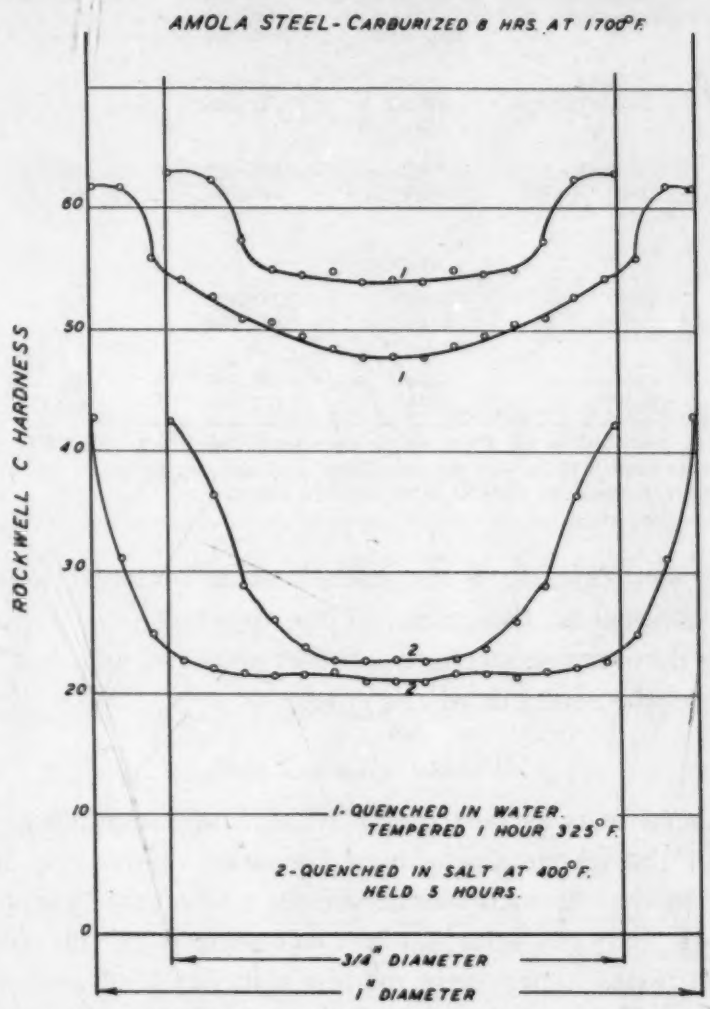


Fig. 16—Steel B. Amola Steel. Carburized 8 Hours at 1700 Degrees Fahr.

of this test and the ease with which different values can be obtained with the same heat treatment if great care is not taken, it appears actually that depth of case has a much greater effect than alloy content.

Both Birchfield and McMullan found that the deeper the case the lower the impact value, and Birchfield suggested that if the area

[4] P. A. Birchfield, "The Effect of Case Depth on the Tensile and Impact Properties of Carburizing Steels." Paper presented before the Northwest Chapter of the American Society for Metals on Jan. 14, 1936. Reported in *The Review*, March 1936.

Table IV
Transverse Bend Tests

Maximum case strength in case hardened specimens—quenched from box after 8 hours at 1700 degrees Fahr. All figures are the average of three specimens.

Steel	Heat Treatment Quenched in	Load Supported in Lbs. at Center of ½ In. Squares 5½ In. Between Supports	Max. Case Strength in P.S.I. Calc. by S = Mc/I	Rockwell Hardness "C" Case	Depth of Case Inches
A					
Plain Carbon	Water[1]	910	68,000	62	0.055
0.64 Per Cent Carbon	Salt[2]	4190	312,000	61	0.055
B					
C-Mo	Water[1]	2900	216,000	64	0.042
0.42 Per Cent Carbon	Salt[2]	3820	285,000	62	0.042
0.20 Per Cent Molybdenum					
C					
S.A.E.	Oil[1]	3940	294,000	60	0.035
2330	Salt[2]	3850	287,000	61	0.035
D					
S.A.E.	Oil[1]	2340	175,000	60	0.055
3120	Salt[2]	2370	177,000	61	0.055

[1]All samples quenched in oil or in water were tempered 1 hour at 325 degrees Fahr.

[2]The salt bath was held at 400 degrees Fahr. and samples of steels A, B and C were held 5 hours while samples of steel D were held 48 hours.

of the core were measured the impact value obtained would be directly proportional to this area. Thus this test was of little value for testing the impact strength of the case and all that has been measured was the strength of the core.

Static Bend Test

Static bend tests as performed by McMullan should give a very good idea of the tensile strength of the case. However, due to the inability to harden ¾-inch rounds of the carbon and carbon-molybdenum steels, bars ½-inch square were made of all four steels for the bend tests. They were made six inches long and cased only on two opposite sides. The bars were supported 5½ inches apart and loaded at the center with the case on top and bottom. The bars were forged to 0.600-inch squares and surface ground on two opposite sides, copper plated and then surface ground on the other two sides to ½-inch squares. Three specimens of each steel were quenched and tempered, while another set of three were given an interrupted quench. The heat treatment was similar to that given the Charpy specimens.

Deflection readings were made on all the steels at intervals of 200 pounds increase in load. The values obtained varied nearly

directly with the load carried. This would be expected with steels having such a high hardness. Only with the nickel-chromium steel was any previous crack obtained before the specimen broke. In all the other tests the specimens broke violently clear across the section.

The results obtained from the bend tests are given in Table IV which shows the great increase of strength in the plain carbon steel

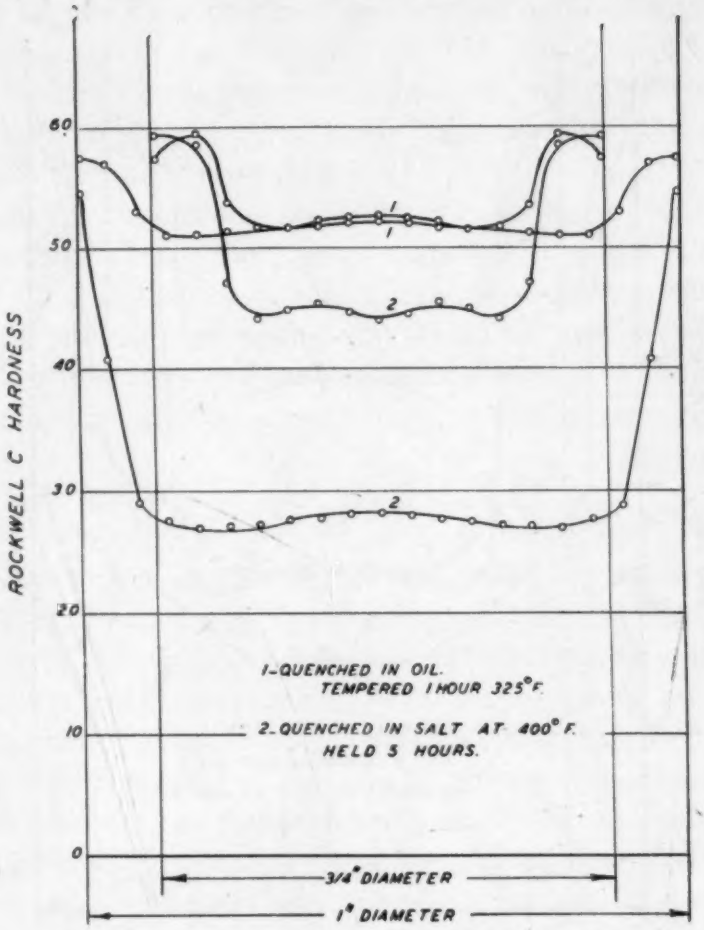


Fig. 17—Steel C. S.A.E. 2330 Carburized 8 Hours at 1700 Degrees Fahr.

when heat treated by the interrupted quench method as compared to the quenched and tempered method. We will admit to begin with that the most suitable heat treatment for maximum strength was not used and pot-quenching from 1700 degrees Fahr. would certainly give low results. However, it was found that the plain carbon steel when quenched in a salt bath had a higher strength than any of the alloy steels and we can probably explain this by the high carbon of the core. This heat treatment has not introduced any stresses in the specimen so that full strength could be developed. In the

case of the specimen quenched in water, the stresses set up between the core and case and possibly between the zones of the case made the specimen unable to carry a very large load. McMullan found that with samples quenched from the pot an increase in case depth decreased their load carrying capacity and in the double treated specimen the increase in case depth did the reverse and increased in strength.

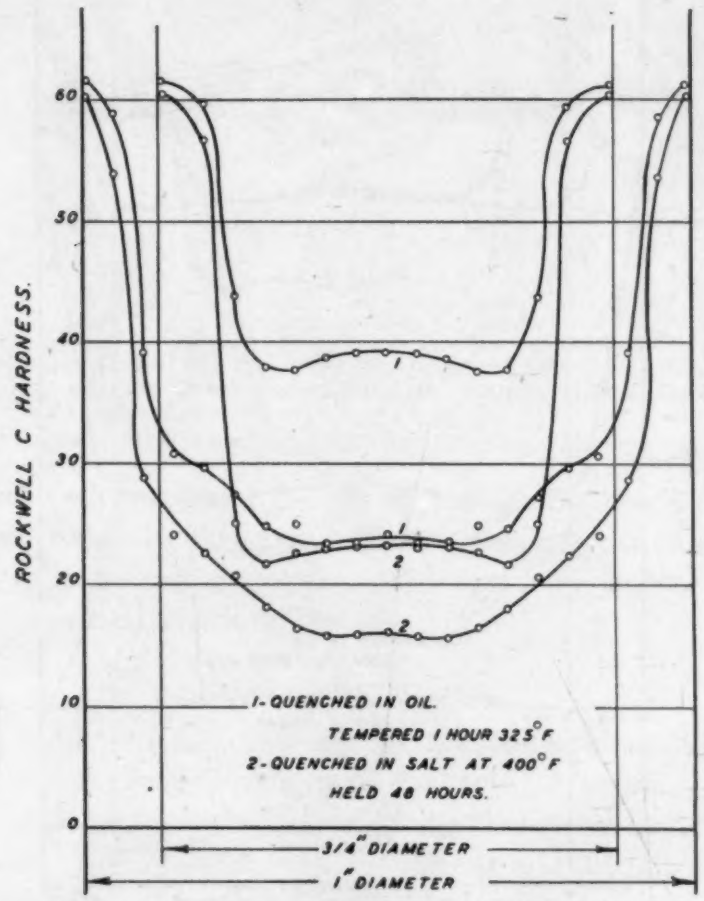


Fig. 18—Steel D. S.A.E. 3120 Carburized 8 Hours at 1700 Degrees Fahr.

The carbon-molybdenum steel showed some improvement by quenching in salt as compared to water, while the nickel and nickel-chromium steels showed no such improvement. This can be explained entirely by the stresses that would be set up in the case of the 0.42 per cent carbon core of the molybdenum steel, while the low carbon core of the other two steels prevents any great amount of stress from occurring due to the softness of the core. The difference in values obtained for the nickel steel over the nickel-chromium steel was mainly due to the higher carbon of the core. Thus we see that, contrary to

usual custom, the core can be very much higher in carbon than has been used, which would result in a much higher strength of the part made. If a high carbon steel is used, however, it must be heat treated by the interrupted quench method in order to eliminate stresses which cause weakness of quenched samples. It would appear that plain carbon steels could be used satisfactorily for all parts, but it must be kept in mind that they can be used only for small parts due to their inability to harden deeply. The introduction of a small amount of chromium or manganese, however, retards the transformation to such a large extent that fairly large cross sections can be heat treated with a relatively low cost steel.

It is interesting to note the hardness obtained by the interrupted quench method. With the Charpy tests a very uniform value of 62 Rockwell C was obtained and in the bend specimens values varying between 61 and 62 were obtained. This indicates very clearly that the hardness resulted from carbide precipitation at 400 degrees Fahr. and increased progressively with time until the reaction was completed.

Conclusions

The hardening of case carburized steels by the delayed transformation of the austenite at temperatures below 600 degrees Fahr. appears to be practical and have several advantages. No improvement in physical properties was obtained by using an interrupted quench on case hardened parts with steels of low carbon content. Since the carbon content of the core of quenched and tempered steels for case hardening has to be low, alloying elements are added to the steel to improve the physical properties. By interrupted quenching, case hardened parts may have a core containing higher carbon in order to increase its strength. The presence of some alloying element is necessary in order to harden large cross sections and for this purpose chromium and manganese seem to be the best adapted because of the low cost and the large effect on the rate of transformation. The cost of the quenching baths, whether of a tempering salt or a lead alloy, will be higher and consequently the total cost of heat treatment will be increased and, with higher carbon steels, it is likely that machining costs will also be increased.

Several practical advantages of this method, however, are as follows:

1. A shallow case would be sufficient to produce surface hard-

ness since the hard core would eliminate the danger of punching through the case, thus reducing the cost of carburizing. In some cases the carburizing step might be eliminated entirely if the stock used were of sufficiently high carbon content.

2. Steels given an interrupted quench do not require any further treatment, thus eliminating many additional steps.

3. Since any stresses set up during transformation are probably relieved as hardening proceeds, with the interrupted quenching process there will be less tendency for distortion, quenching cracks and grinding cracks.

The interrupted quenching process described is far more simple than would at first appear. A test is made to determine whether the steel selected will harden in the cross section required and the time is determined for complete transformation of the steel at the temperature of the tempering salt or lead alloy bath. This temperature depends on the hardness desired and the length of time required for transformation. Ordinarily, for hardness of 60 Rockwell C or over, the salt bath temperature will be 400 degrees Fahr. for a period of 5 hours or more depending on the carbon and alloy contents. For a hardness of 55 Rockwell C the salt bath temperature will be 600 degrees Fahr. and the time will be reduced to about one hour. It is not necessary to transform completely in the salt bath but the sections can be removed after attaining salt bath temperature and be transferred to any other furnace at the same temperature in order to economize on salt bath capacity. The quenching temperature used in this work was 1700 degrees Fahr. but lower quenching temperatures should give about the same results.

It is hoped that these experiments will serve as a guide in the development of special low alloy steels of high carbon content which will give better service in industry when treated by the interrupted quenching method.

ACKNOWLEDGMENT

The authors gratefully acknowledge their indebtedness to E. C. Bain, E. S. Davenport and R. H. Aborn of the U. S. Steel Corporation. The steels were furnished by Walter Mathesius and E. T. Barron of Carnegie-Illinois Steel Corp.

Thanks are also extended to all members of the Department of Metallography and to other staff members of the University; namely, Professors T. P. Hughes, G. C. Priester, F. E. Miller, and W. E. Apuli, for aid in this work.

THE PRODUCTION OF FLAKES IN STEEL BY HEATING IN HYDROGEN

By R. E. Cramer

Abstract

A brief review of the literature shows that no conclusive proof of factors affecting the development of flakes was presented until 1935. In that year work reported from Germany and Italy showed that steel treated in hydrogen would develop flakes.

Flaked and sound steel was analyzed for gases by the Vacher-Jordan vacuum fusion method. No relation between gas content and flakes or between microstructure and flakes was established.

Photographs are presented showing: A fissure developed from a flake in a locomotive tire; flakes in steel as received from the mill, both in segregation streaks, and scattered at random; and flakes and cracks developed in 1½ x 3 x 10-inch steel bars heated in hydrogen at 2300 degrees Fahr. (1276 degrees Cent.) for 27 hours and cooled in various ways.

A discussion is presented on the possible mechanism of the development of flakes and the sources of hydrogen in the open-hearth furnace. The conclusion is drawn that hydrogen is one factor which will produce flakes in steel.

The Production of Flakes in Steel by Heating in Hydrogen

MOST metallurgists are familiar with the problem of flakes in steel through the numerous technical articles on the subject. Ashdown (1)[1] has furnished one of the best sets of photographs of such flakes. The accompanying photograph, Fig. 1, taken by the writer in 1924 shows a rather large flake ⅜ inch under the bearing surface of a locomotive tire. The flake is oval in shape and rather coarse-grained. Starting at the left edge of the flake a smooth bright internal fissure has developed by fatigue action,

[1]The figures in parentheses refer to the bibliography appended to this paper.

A paper presented before the Eighteenth Annual Convention of the Society, Cleveland, October 19 to 23, 1936. The author, R. E. Cramer, is Special Research Assistant in Engineering Materials, Engineering Experiment Station, University of Illinois. Manuscript received July 25, 1936.

extending over half the width and thickness of the tire. Final failure of the remainder of the metal was by sudden fracture, producing a coarse grain appearance.

For over fifteen years the question of flakes in steel baffled the best metallurgical minds who sought a satisfactory explanation. Many hypotheses of the cracking of steel as it cools in open air to room temperature after hot working have been offered with some supporting evidence. However, until 1935 no convincing evidence was offered that any controlling factor which influences the development of internal flakes or cracks, had been discovered.

Results from European Laboratories

In March 1935 Bennek, Schenck and Müller (2) in one paper and Houdremont and Korschan (3) in another announced results showing that hydrogen would cause flakes in steel when molten steel is exposed to hydrogen gas. This work together with other information on gases in metals was ably abstracted by Epstein (4). In November 1935 Musatti and Reggiori (5) showed that, when heated in hydrogen at 2100 degrees Fahr. (1150 degrees Cent.) for a number of hours, round bars of both alloy and carbon steel, 2 to 3 inches in diameter, contained flakes when air cooled to room temperature. Their work was recently published in METAL PROGRESS (6) and includes the results of analyses of gas in the steel. It is interesting to note from their results that the steel cooled in air lost practically all its hydrogen, while the steel cooled in water retained a good portion of it.

Search for the Cause of Flakes

The writer of this paper began work on the problem of flakes in steel in 1931. Statistical evidence as to the prevalence of flakes was accumulated on 175 heats of open-hearth steel with carbon contents ranging from 0.69 to 0.84 per cent and manganese contents ranging from 0.51 to 0.90 per cent. Some flakes were found in steel from about 25 per cent of the heats. Many specimens were examined under the microscope, but no correlation was established between crystalline structure and flakes. In certain heats flakes were especially numerous in segregation streaks, but in many cases similar segregation streaks were free from flakes. Fig. 2 shows several transverse and longitudinal flakes revealed by deep etching which

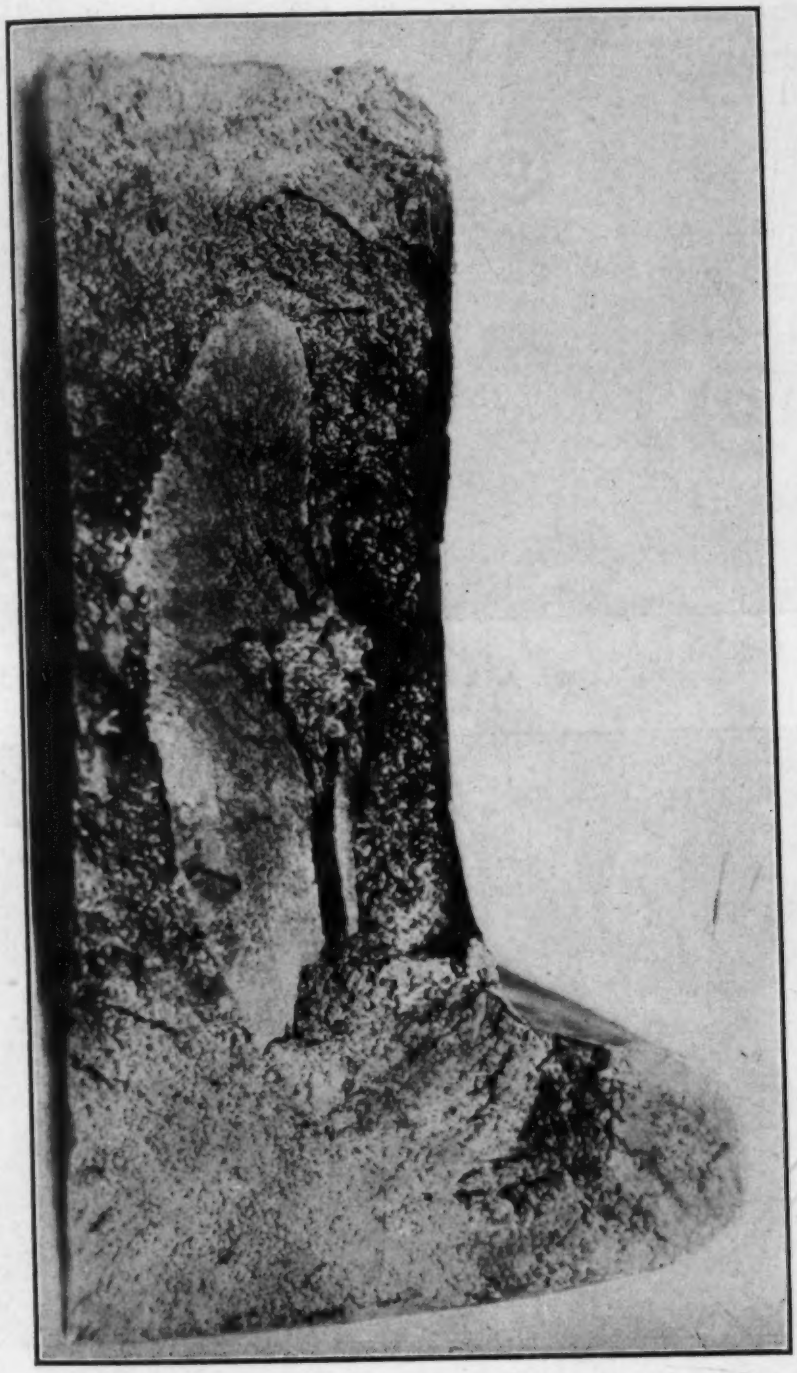


Fig. 1—Flake and Fissure in Locomotive Tire. This Illustration is Approximately the Actual Size of the Specimen.

are confined entirely to two segregation streaks. Fig. 3 shows another etched specimen with so many flakes that it is obvious that they could not all have developed in segregation streaks.

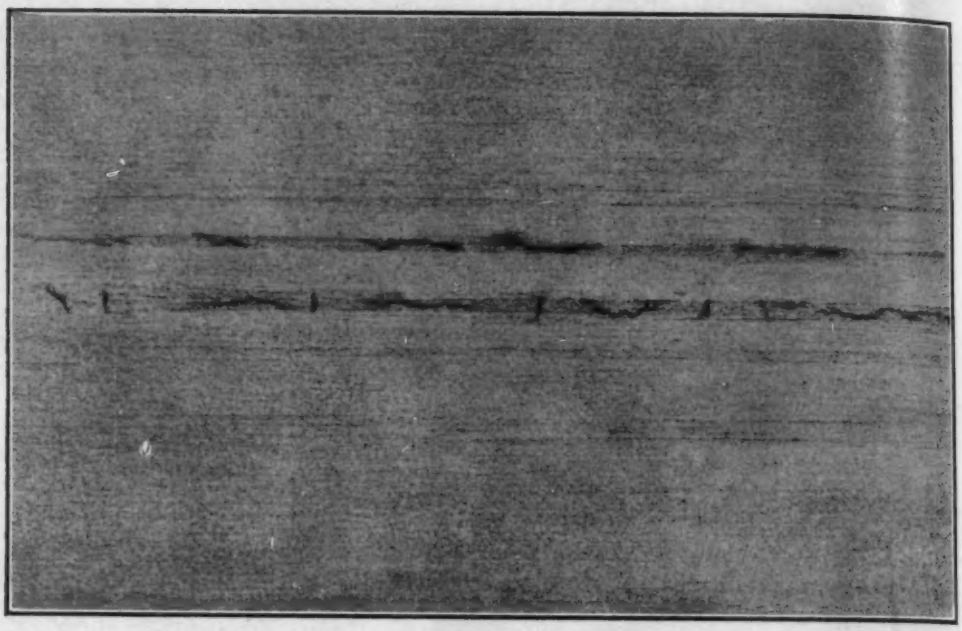


Fig. 2—Flakes Confined to Segregation Streaks. Approximately Natural Size. Etched in Hot 50 Per Cent Hydrochloric Acid.

Fig. 3—Flakes Appearing at Random in Steel Bar. Approximately Natural Size. Etched in Hot 50 Per Cent Hydrochloric Acid.

Analysis of Gases Remaining in Flaked Steel

After considering the various possible causes of flakes it was decided to make analyses of steel with flakes and steel without flakes for the presence of three gases, (1) nitrogen, which had been sug-

gested by Dean (7) as a possible influence in developing flakes, (2) oxygen, which was blamed as the cause of many troubles in steel, and (3) hydrogen, which had been suggested to the writer by a teacher of metallurgy as a possible cause of flakes. Since the vacuum fusion method of analysis developed by Vacher and Jordan (8) could be used to determine all three of these gases, work was started on the gas analysis of flaked and non-flaked bars of steel, using this method. The mill analyses of the bars so studied are given in Table I. The gas analyses of the bars, together with that of an analyzed sample furnished by the Bureau of Standards are given in Table II. The results of these analyses were disappointing in that they did not show any realationship between flakes and the gas content of the steel. More recent results which have been reported also confirm this conclusion. There is a rather close agreement in oxygen and nitrogen content of bars from the same mills.

Laboratory Production of Flakes in Sound Steel

Following the example of work done in German laboratories, experiments were next started in which molten steel was treated

Table I
Mill Analyses of Steel Bars

Flakes in Bar	Lab. No. of Bar	Mill	Chemical Composition—Per Cent				
			Carbon	Manganese	Silicon	Phosphorus	Sulphur
Yes	507	D	0.83	0.78	0.29	0.023	0.030
Yes	1005	L	0.68	0.84	0.17	0.020	0.040
Yes	430	A	0.76	0.58	0.27	0.015	0.024
No	561	A	0.77	0.54	0.22	0.019	0.024
No	482	D	0.85	0.90	0.21	0.025	0.027

Table II
Gas Analysis of Steel Bars and of Standard Samples

Flakes in Bar	Lab. No. of Bar	Mill	Gas Content—Per Cent		
			Oxygen	Nitrogen	Hydrogen
No	Std. from Bureau of Stds.		0.077	0.0061	.....*
No	Std. from Bureau of Stds.		0.087	0.0072	.....*
No	Std. from Bureau of Stds.		0.088	0.0072	.....*
No	Std. from Bureau of Stds.		0.085	0.0060	.....*
	Av. analysis at U. of I.		0.085	0.0066	.....
	Analysis reported by Bureau of Standards		0.075	0.005	0.0004
Yes	507	D	0.0015	0.0016	.....*
Yes	507	D	0.0012	0.0017	.....*
Yes	1005	L	0.0023	0.0013	.....*
Yes	1005	L	0.0027	0.0018	.....*
Yes	430	A	0.0028	0.0023	.....*
	Average of flaked bars		0.0022	0.0018	.....*
No	561	A	0.0032	0.0039	.....*
No	561	A	0.0038	0.0037	.....*
No	482	D	0.0016	0.0023	.....*
No	482	D	0.0017	0.0021	.....*
	Average of bars without flakes		0.0026	0.0030	.....*

*The amount of hydrogen in all bars was found to be so small as to be less than the range of accuracy of the analytical method used at the University of Illinois.

with hydrogen gas. These were abandoned because of the dangers involved when working with hydrogen in an unventilated room. The next method of approach consisted in heating bars of steel about 1½ x 3 x 10 inches long in an atmosphere of hydrogen. This method was less dangerous and proved successful. The composition of the steel in which flakes have been produced by this method fell within the following limits:

	Per Cent
Carbon	0.71–0.80
Manganese	0.70–0.80
Silicon	0.20–0.29
Phosphorus	0.019–0.022
Sulphur	0.024–0.041

Before any test specimen was heated in hydrogen, short specimens were cut from the same bar of steel immediately adjacent to the ends of the test specimen, and it was ascertained by deep etch tests of these short specimens that the bar before heating in hydrogen was free from flakes. The test specimen was then heated in a carbon-pile electric furnace with hydrogen gas flowing through the furnace at atmospheric pressure at the rate of 10 cubic feet of gas per hour. The temperature of the furnace was in most cases held between 2200 and 2300 degrees Fahr. (1200-1260 degrees Cent.) for 27 hours. All specimens which developed typical flakes were cooled in still air or under cooling fans. One specimen was quenched in water after cooling to 1500 degrees Fahr. (815 degrees Cent.) in air. Only one specimen heated in hydrogen failed to contain flakes, and in this case the steel was heated to a temperature below 2200 degrees Fahr. (1200 degrees Cent.). Check runs were made by heating specimens from the same bars to 2300 degrees Fahr. (1260 degrees Cent.) for 27 hours without the current of hydrogen gas, then cooling in still air. No flakes developed in the specimens not heated in hydrogen.

Figs. 4 and 5 show typical flakes produced in specimens heated in hydrogen and cooled in air. These flakes are somewhat larger than but not so numerous as the flakes in some bars of steel received from steel mills. (See Figs. 2 and 3.) One specimen heated in hydrogen was heated above 2300 degrees Fahr. (1260 degrees Cent.), the exact temperature not being known, but to a temperature at which the surface of the steel started to melt. The cracks found in this specimen after cooling in air are shown in Fig. 6. They appear to be at the austenite grain boundaries. This is an exceptional result which has not yet been duplicated, and which calls for further study.

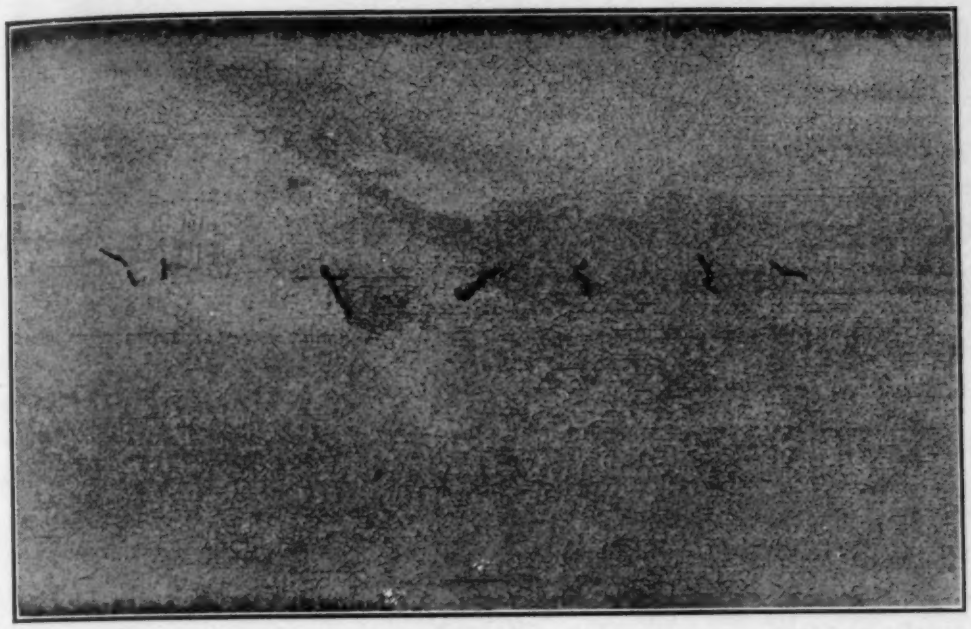


Fig. 4—Flakes Produced by Heating Sound Steel at 2200 Degrees Fahr. (1200 Degrees Cent.) in Hydrogen for 27 Hours and Cooling in Air. Approximately Natural Size. Etched in Hot 50 Per Cent Hydrochloric Acid.

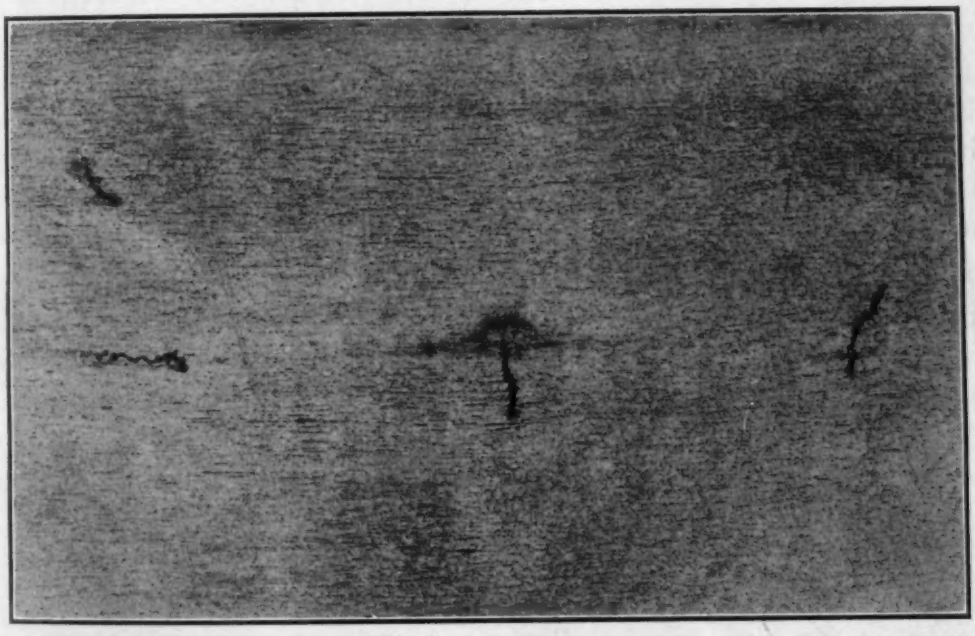


Fig. 5—Flakes Produced by Heating Sound Steel at 2300 Degrees Fahr. (1260 Degrees Cent.) in Hydrogen for 27 Hours and Cooling in Air. Approximately Natural Size. Etched in Hot 50 Per Cent Hydrochloric Acid.

Fig. 7 shows cracks developed in the specimen heated in hydrogen and quenched in water. This bar developed two quenching bursts extending nearly across the specimen, but not coming to the surface at any point. As can be seen, the specimen contains several

hundred short hairline cracks, or flakes, on both sides of one burst, but is practically free of these short cracks within half an inch of the burst. If the absence of cracks near the burst is due to the diffusion of hydrogen over to the edge of this half-inch space, this diffusion must take place very rapidly, since the burst was plainly heard during the quench, occurring after the rapid evolution of steam. It seems reasonable to assume that cooling stresses played some part in the production of the small cracks, and that the relief of these stresses in the metal adjacent to the burst accounts for their absence in this half-inch area.

The appearance and distribution of these small cracks in the quenched steel remind one of the microscopic cracks reported by Davenport, Roff and Bain (9) except that the latter were of extremely small size. Also both types of cracks are reported to develop at a temperature of about 400 degrees Fahr. (200 degrees Cent.).

In a previous discussion (10) the writer cited a possible explanation of why the cracks develop at a temperature of about 400 degrees Fahr. (200 degrees Cent.). The explanation is based on a report that there is a sudden drop, during cooling, in the rate of diffusion of hydrogen through steel at this temperature. This change in rate of diffusion of hydrogen through iron is confirmed this year by Ham (11), although his data (Fig. 13, page 11) indicate that it occurs over the temperature range of 400 to 475 degrees Fahr. (205 to 245 degrees Cent.). It is interesting to note that this is the location of the A_o line in the iron-carbon equilibrium diagram as published by the American Society for Metals. Further tests to determine more accurately the temperature at which flakes develop are contemplated.

Possible Explanation of Development of Flakes

While the mechanism of the production of flakes is not thoroughly understood, the following explanation, which was partly presented by the European metallurgists, appears to agree with the available information.

The molten steel in the open-hearth absorbs more than its own volume of hydrogen. During cooling of the steel some hydrogen escapes, but the steel holds a good portion of it in solution until it cools below the critical point after rolling. The rate of cooling of the steel in open air is not so fast but that hydrogen can diffuse out

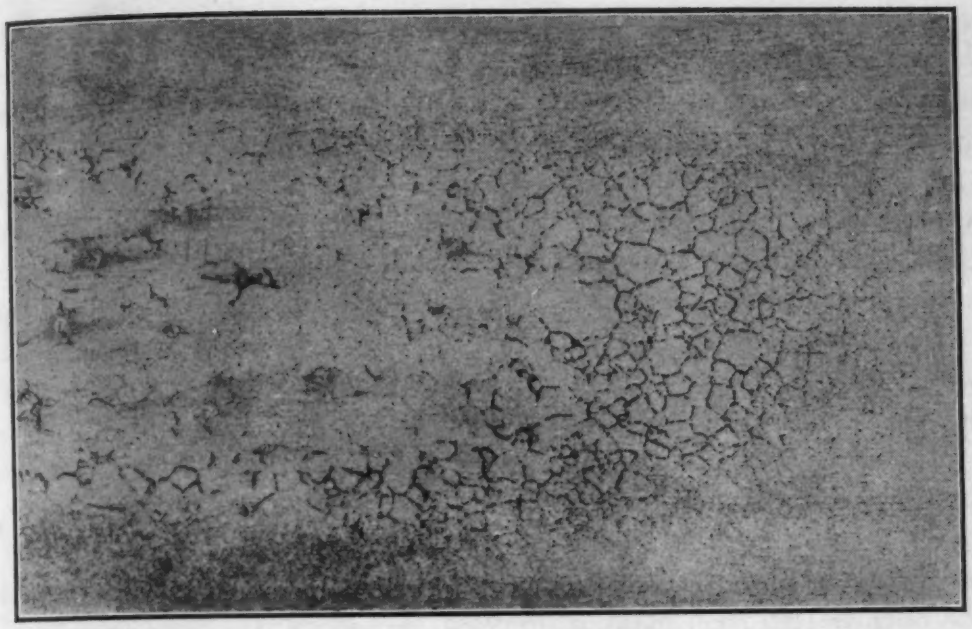


Fig. 6—Cracks Produced in Sound Steel by Heating in Hydrogen for 27 Hours at a Temperature at Which the Surface Started to Melt. Cooled in Air. Approximately Natural Size. Etched in Hot 50 Per Cent Hydrochloric Acid.

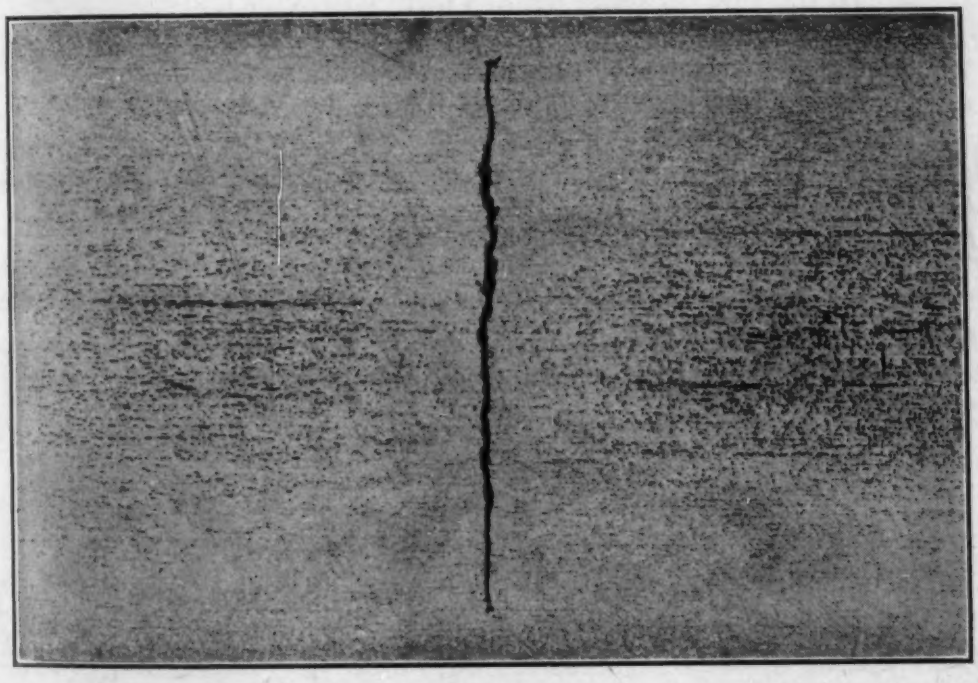


Fig. 7—Cracks Produced in Sound Steel by Heating at 2300 Degrees Fahr. (1260 Degrees Cent.) in Hydrogen for 27 Hours, Cooling in Air to 1500 Degrees Fahr. (815 Degrees Cent.), then Quenched in Water. Approximately Natural Size. Etched in Hot 50 Per Cent Hydrochloric Acid.

of the steel as it cools down to some temperature in the neighborhood of 400 degrees Fahr. At this temperature there is still in the steel a

considerable volume of hydrogen, which is being liberated due to the lowering of the solution pressure accompanying the drop in temperature. When the diffusion rate suddenly changes the hydrogen which is being liberated is trapped in the interior of the steel, and probably collects in minute spaces around inclusions and in any other small voids. The pressures built up in these small spaces must be very high, and, since the stresses in the metal are distributed over minute areas, the combination of hydrogen pressure and cooling stresses may exceed the tensile strength of the steel. A small crack then develops which relieves the stresses. The crack remains in the cooled steel as a flake.

This explanation calls for some consideration as to how hydrogen can be introduced into the molten steel. There are many ways in which water, which, when dissociated, produces two volumes of hydrogen to one of oxygen, may be introduced or produced during the open-hearth steel making process. Some sources are:

1. Production during the combustion of the fuel in the open-hearth furnace.
2. In the rust on scrap metal (iron hydroxide).
3. In the molten metal from the blast furnace.
4. In the ore and the lime used in the open-hearth furnace.
5. In the fuel and the air used for combustion.

The first item accounts for the largest supply of water. At the temperature of the open-hearth furnace the slag and the molten steel could readily break water vapor up into oxygen and hydrogen, through the reaction $Fe + H_2O = FeO + 2H$. The hydrogen would be readily absorbed by the molten steel. During the course of the production of a heat of open-hearth steel there seems to be a considerable opportunity for the absorption of hydrogen produced from the dissociation of water vapor.

Summary and Conclusions

It has been shown that flakes or small cracks are produced in an 0.80 per cent carbon, 0.80 per cent manganese crack-free steel when heated at 2200 degrees Fahr. (1200 degrees Cent.) for 27 hours in an atmosphere of hydrogen, followed by cooling in air. A different type of cracking resulted when a specimen was heated in hydrogen until the surface began to melt, and then air cooled. Specimens heated in hydrogen and then quenched in water showed numerous small cracks.

Although it has been shown that hydrogen can cause flakes in steel during cooling, this is no proof that other causes, such as localized temperature gradients, cannot also cause flakes.

Gas analyses of flaked steel have failed to show any relation between the presence of flakes and the gas content of the steel after cooling.

Further experiments are necessary to determine accurately the temperature at which flakes are formed.

Acknowledgments

The work reported in this paper has been done under the direction of Professors H. F. Moore and H. R. Thomas of the staff of the Engineering Experiment Station of the University of Illinois. The vacuum fusion analyses were made under the direction of Dr. G. F. Smith of the Department of Chemistry of the University of Illinois. The writer wishes to express his appreciation for their help and encouragement throughout the work.

Bibliography

1. H. H. Ashdown, "Prevention of Flakes in Alloy Steels," Metal Progress, Vol. 24, November 1933, p. 13.
2. H. Bennek, H. Schenck and H. Müller, "Die Entstehungsursache der Flocken im Stahl," *Stahl und Eisen,* Vol. 55, March 21, 1935, p. 321.
3. E. Houdremont and H. Korschan, "Die Entstehungsbedingungen der Flocken im Stahl," *Stahl und Eisen,* Vol. 55, March 14, 1935, p. 297.
4. S. Epstein, "Recent Progress in Steel Making Reported from Germany," *Metals and Alloys,* Vol. 6, June 1935, p. 160; July 1935, p. 181; August 1935, p. 219.
5. I. Musatti and A. Reggiori, "Quelques Remarques Sur la Formation des Flocons dans les Aciers au Chrome-nickel-molybdene," *Revue de Metallurgie,* Vol. 32, November 1935, p. 531.
6. I. Musatti and A. Reggiori, "Flakes in Forgings Caused by Hydrogen," Metal Progress, Vol. 30, July 1936, p. 51.
7. R. S. Dean, "The Absorption of Nitrogen by Steel," Report of Investigations, U. S. Bureau of Mines, Number 3076, March 1931, p. 3.
8. H. C. Vacher and L. Jordan, "The Determination of Oxygen and Nitrogen in Irons and Steels by the Vacuum Fusion Method," U. S. Bureau of Standards, Research Paper No. 346, 1931.
9. E. S. Davenport, E. L. Roff and E. C. Bain, "Microscopic Cracks in Hardened Steel, Their Effects and Elimination," Transactions, American Society for Metals, Vol. XXII, April 1934, p. 289.
10. R. E. Cramer, "Shatter Cracks and Flakes in Steel," *Metals and Alloys,* Vol. 7, January 1936, p. 23.
11. W. R. Ham, "The Diffusion of Hydrogen Through Nickel and Iron," Transactions, American Society for Metals, Vol. 25, 1937, p. 536.

DISCUSSION

H. H. ASHDOWN:[1] I personally cannot accept the hydrogen theory as I cannot conceive of hydrogen in the atomic form being residual in the steel after being slowly heated to forging temperature and being so retained after forging.

From my own experience during electro-deposition plating I have seen steel embrittled by hydrogen and by heating in glycerine to around 350 degrees Fahr. have seen the hydrogen slowly liberated and the steel return to its normal ductile condition. It is now well known that flakes existing in steel billets will weld up on reforging but, if the billets are again cooled in air to normal temperature a fresh crop of flakes, usually smaller, will develop.

If hydrogen were the sole cause of flake—according to the procedure outlined in recent published researches and from the instance cited above, by slowly reheating these billets to forging temperature the hydrogen should be dispersed and the billets on air cooling should be free from flake.

Further, large ingots and billets which for commercial reasons have to cool to room temperature—although cooled exceedingly slowly and subsequently, for forging, are very slowly reheated, first, to about 1200 degrees Fahr. then, raised to forging temperature, will produce forgings full of flake if allowed to cool in air. Some of these ingots are trepanned to a large bore prior to heating, for making hollow forgings and thus should provide free passage for the diffusion of hydrogen. Such result does not appear to support the experimental data in recent research publications on this subject.

It has already been proven that the higher the temperature a forging is raised above the Ac normal, the lower will the Ar transformation be depressed if the forging is cooled in air, bringing this point well within the austenitic-martensitic transformation embrittling range. It is not uncommon for ingots to be stripped as soon as they are self-supporting and transferred to soaking pits or forge furnaces. Forgings made from such ingots are actually cooling from the temperature of liquid steel to the Ar transformation, where high expansion stresses may result due to volume change. All these facts are, together with the possible presence of hydrogen, deserving of careful consideration. Until the cause of flake is more definitely known and methods of manufacture adopted for its correction, these defects can be prevented by a suitable heat treatment to the finished forging.

Author's Closure

Mr. Ashdown presents many good arguments against hydrogen being the cause of flakes, which the speaker cannot answer. His point on the lowering of the Ar transformation to the temperature range where flakes are reported to develop seems quite logical for certain alloy steels but I do not believe it applies to the plain carbon steel used in these experiments. Cooling curves have been run on a number of bars which developed flakes on cooling in air from the rolling temperature and the Ar transformation always occurred around 1250 degrees Fahr. (680 degrees Cent.). No cooling curves were run on the specimens heated in hydrogen at 2300 degrees Fahr. (1275 degrees Cent.).

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[1]Consulting metallurgist, 901 Hill Ave., Pittsburgh.

DIFFERENTIAL HARDENING BY INDUCTION

By M. A. Tran and W. E. Benninghoff

Abstract

The development and growth of modern day automotive transportation has imposed severe operating conditions on all parts of the vehicle and especially the crankshaft. To meet the new conditions a different approach to the problem was made by the application of high frequency inductive heating to harden only the wearing surfaces without affecting the core of the part which had been previously heat treated to meet the physical requirements. The new process of differential heating by induction employs an average heating cycle of only a few seconds and produces a hardened area accurately controlled as to structure, depth, width and location. The resulting surface structure of high hardness with its gradual blending into the core structure is described along with suggested changes and modifications in crankshaft production procedure.

THE growth of inter-city bus and truck service is in a way a measure of the development of the automotive heavy-duty engine and especially of the crankshaft. The earlier engines were equipped with heat treated carbon steel crankshafts, but serious difficulty was experienced with them due to bearing wear, as the size of loads and operating periods increased.

In order to obtain harder, more wear-resistant bearing surfaces, the engine designers then resorted to alloy steels, heat treating them up to the limit of machinability. These shafts, of course, showed a marked improvement in bearing wear. As motor development progressed and with the advent of the automotive-type Diesel engine with higher bearing speeds and pressures, new bearing materials such as copper-lead, cadmium-silver and cadmium-nickel were introduced to meet the new demands. The use of these harder bearing materials led to excessive crankshaft wear, and there arose an insistent demand for still better wear-resistant bearing surfaces to meet the new conditions. In other words, the motor designer, the

A paper presented before the Eighteenth Annual Convention of the Society held in Cleveland, October 19 to 23, 1936. Of the authors, M. A. Tran is chief metallurgist, Park Drop Forge Co., and W. E. Benninghoff is Tocco Division manager, Ohio Crankshaft Co., Cleveland.

truck and bus salesman, and the fleet owners demanded a crankshaft that would be capable of operating many thousands of miles without a bearing failure or even a bearing adjustment.

Carburizing, nitriding, and many other methods of surface hardening were tried to produce the desired results. All of these methods were found wanting in one particular or another and they all require the addition or absorption of other elements to the surface to be hardened. Some require time cycles as long as 90 hours. The use of high temperature or long time cycles, or both, usually causes excessive distortion. This excessive distortion, of course, requires straightening before machining. In some cases, due to the hardness penetrating the fillets, it has been found impossible to straighten without cracking the shaft in the fillets. Another drawback of distortion is that grinding costs are increased, since more stock must be left for grinding to compensate for some of the distortion. Furthermore, it is found that grinding checks often occur due to the lack of uniformity of structure and case depth. All of the previous methods were affected more or less by variables which were difficult to control and thus made it difficult to consistently reproduce results.

A fair degree of success in surface hardening crankshafts by the flame method has been reported in Europe. An acetylene torch with a fan-shaped burner tip plays the flame on a journal as it is rotated slowly, followed by pressure quenching directly beneath the burner tip. The prime objection to this method is that the resulting hardened layer has a soft area on the surface parallel to the axis where the flame in completing one revolution overlaps the hardened area previously produced.

A further development in this country of the same process is the use of an improved burner with several tips with staggered orifices, together with baffling. The shaft is rotated rapidly on centers, and the entire journal is heated before quenching.

The objection to this method as employed in the United States is that the excessive amount of heat employed in heating the entire bearing before quenching results in excessive heating in the fillets, thereby destroying all previous heat treatment, and causing excessive distortion of the crankshaft. It is difficult to control the flame temperature with any degree of accuracy so that duplication of structure and of the dimensions of the hardened area cannot be maintained, and there is also the constant danger of over-heating.

A method was needed that would produce the desired surface hardness with a structure which would withstand wear and abrasion without being subject to flaking or spalling. It must be accurate as to control of the depth, width, and location of the hardened area and give absolute uniformity of hardening from bearing to bearing and crankshaft to crankshaft. It must have a short time cycle in order to meet production requirements, and the method must not be expensive.

If such a method were developed, a definite contribution to industry and the art of treating metals would be made. We believe such a method has now been developed and it is in the description of this method and the results obtained, particularly on crankshaft bearings, that we are here concerned.

The ideal method for producing a hard surface on a tough core without the addition of carbon or other elements would be to select the proper steel and locally heat the surface rapidly above the Ac_3 point to a desired depth and then quench. The difficulty in such selective surface heating was experienced in obtaining absolute control of the temperature at the location and to the depth desired. Heating must be extremely rapid to prevent undesirable heating of the adjacent areas, especially the fillets.

Dr. E. F. Northrup, of the Ajax Electrothermic Corp., in recent years has done a great deal of preliminary work in investigating the induction heating of solids and the process herein described is based partly on the results of his investigation.

The New Method

High-frequency current of 2000 cycles is passed through an inductor block which surrounds but does not touch the bearing to be hardened. This high-frequency current produces a strong magnetic field which cuts the bearing surface through a small air gap, and induces eddy currents in the bearing surface. The magnetic field also creates hysteresis losses in the bearing surface. The combined eddy current and hysteresis losses, therefore, cause heat to be generated in the surface of the steel itself. The inductor block remains comparatively cold. Fig. 1 shows the focus inductor coils used in producing the hardened specimens. (Note the holes in inductor block which are used for water quench.) Fig. 2 shows the vertical type of induction heating and hardening unit for production hardening of crankshafts.

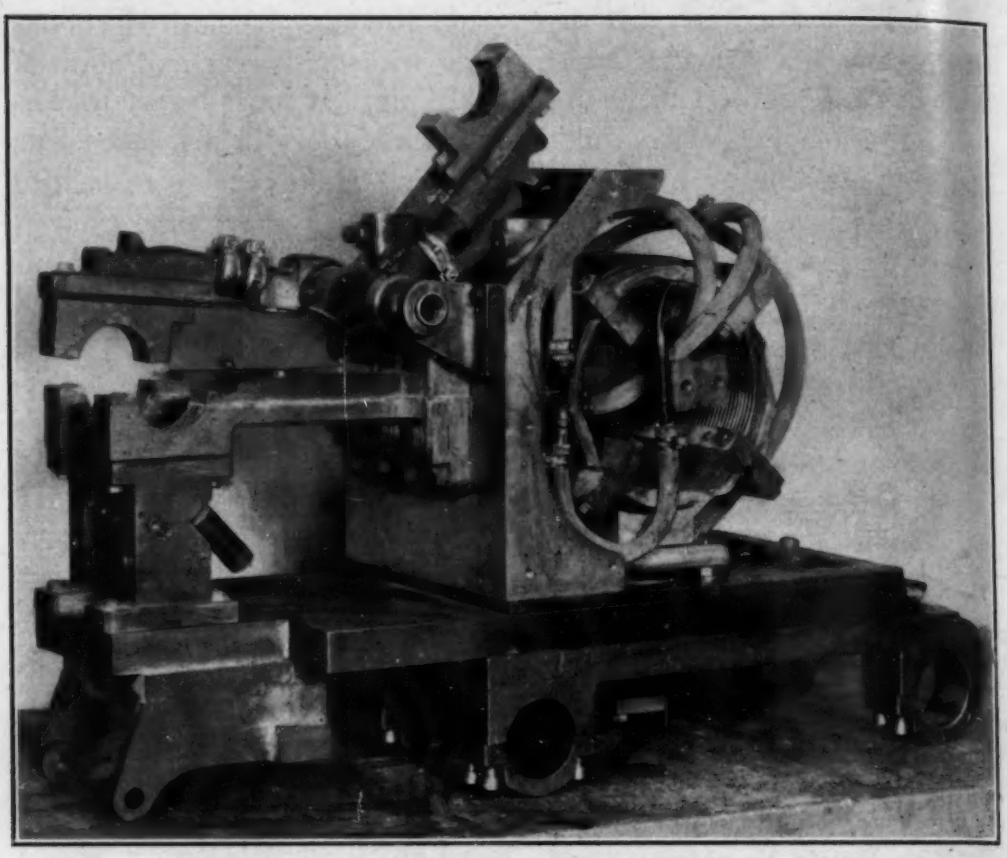

Fig. 1—Photograph Showing the Focus Inductor Coils Used in Producing Hardened Specimens. The Holes in the Inductor Block are Used for Water Quenching.

Due to the inherent reaction of the steel as its temperature rises, the heating effect decreases as the critical point of the steel is approached and, consequently, the surface of the steel is not overheated since continued application of power would only cause the heat to be generated farther in toward the center of the shaft. The mathematics of this heating is quite involved since it is dependent upon the design of the inductor block, the strength of the magnetic field, the frequency of the current, the width of the air gap between the block and the shaft, the steel analysis, the grain size of the steel, the temperature of the steel, as well as the electrical resistance and permeability of the steel.

With the application of high-frequency current to the inductor block, the bearing surface is heated to the quenching temperature for a depth of approximately ⅛ inch in approximately 5 seconds, and is then immediately quenched by pressure-spraying through orifices drilled through the inductor blocks and into a water jacket which is integral with the block. Fig. 3 shows a transverse disk of

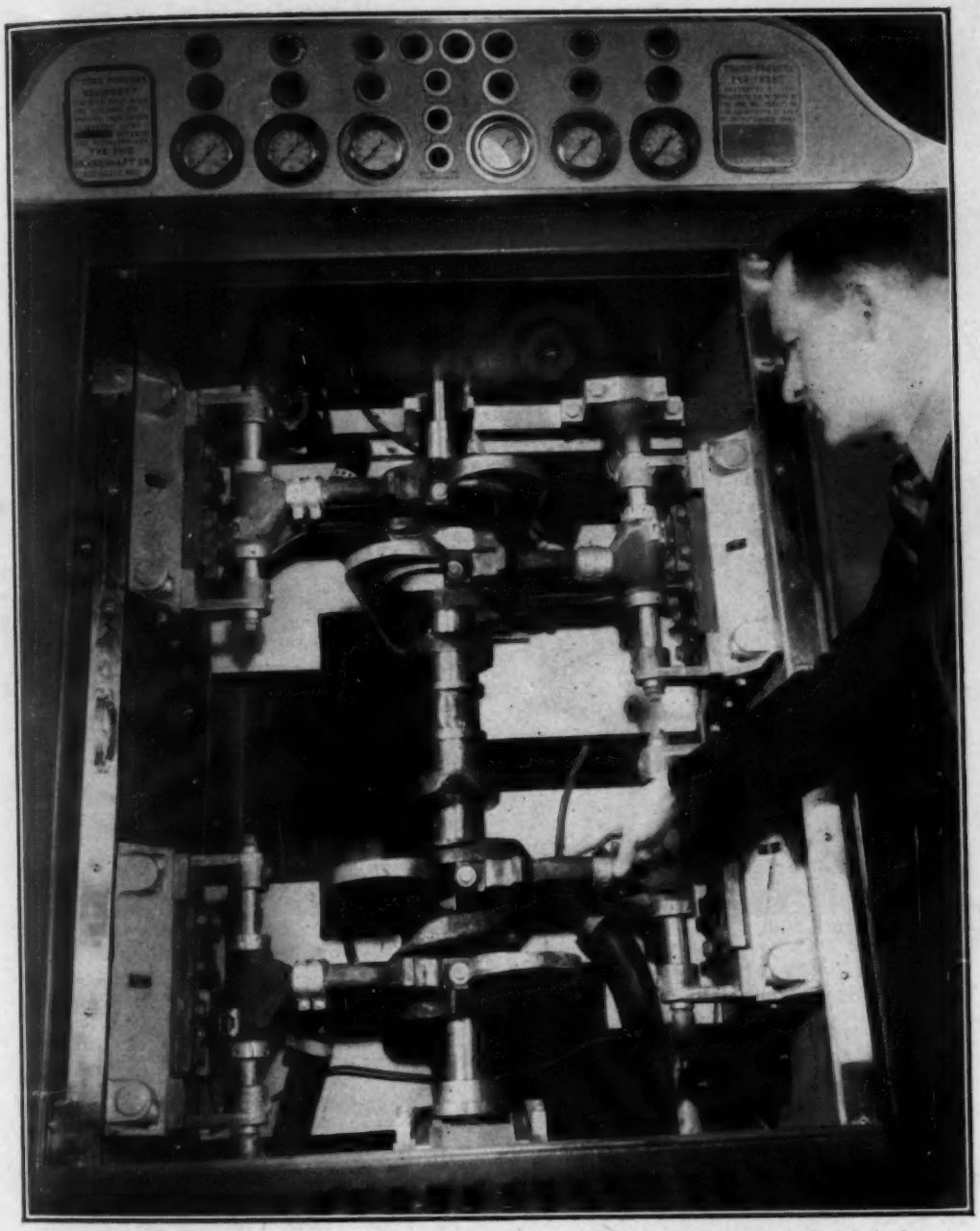

Fig. 2—Vertical Type Hardening Unit for the Production of Hardened Crankshafts.

a Diesel engine crankshaft bearing showing the uniformity obtained on a 3⅝-inch diameter. Fig. 4 shows the longitudinal section of a pin bearing, illustrating the absolute control of the process in regard to width. (Note the complete absence of heating in the fillets.) Fig. 5 is a longitudinal section of a refrigerator compressor crankshaft, in which the thrust wall has been purposely hardened, leaving the included fillet untouched. This illustrates the extreme accuracy

Fig. 3—Photograph of a Transverse Disk of a Diesel Engine Crankshaft Showing the Uniformity Obtained on a 3⅝-Inch Diameter.

of the process. As an example of the speed with which the heating is accomplished on this particular shaft we have the following dimensions:

Front main bearing .....1¼" rd. x 1⅜" long
Rear main bearing .....1¼" rd. x 1¼" long
Pin main bearing .....1¼" rd. x 1½" long
Average heating time —4 seconds per bearing.

The rate of power input and length of time that it is applied vary with different sizes of bearings as well as with different steels. The kilowatt-seconds required for a particular bearing are calculated from graphs compiled from previous data, with due allowance being made for the mass effect of adjacent areas such as crank cheeks. This calculation is then checked by experimentation.

Due to the speed of the cycle, which is measured in split sec-

onds, there is no scaling or pitting of the bearing surfaces, which remain practically as clean as they were before surface hardening. Furthermore, since only the bearing surfaces are heated, there is little chance for distortion of the shaft. All of the control factors, such as rate of power input, length of heating cycle, quenching pressure, and time of quenching are automatic. The operator's only function is to place the shaft in position and push a button to start the cycle. Once the control has been set to produce a certain result on a certain piece, all the pieces will be absolute duplicates in the heat treatment given them.

Fig. 4—Photograph of a Longitudinal Section of a Pin Bearing, Illustrating the Control of the Process in Regard to Width. Note Complete Absence of Heating in the Fillets.

Although the general principles of induction heating have been known for some time, they had not been applied in the field of surface hardening because the accuracy of control necessary for the successful application of high-frequency induction heating had not been developed. The development of the necessary technique of control of all hardening and quenching factors as well as the development of necessary electrical and mechanical equipment has made practical the differential hardening by high-frequency induction and is leading to its general adoption by industry.

The process of differential hardening by high-frequency induc-

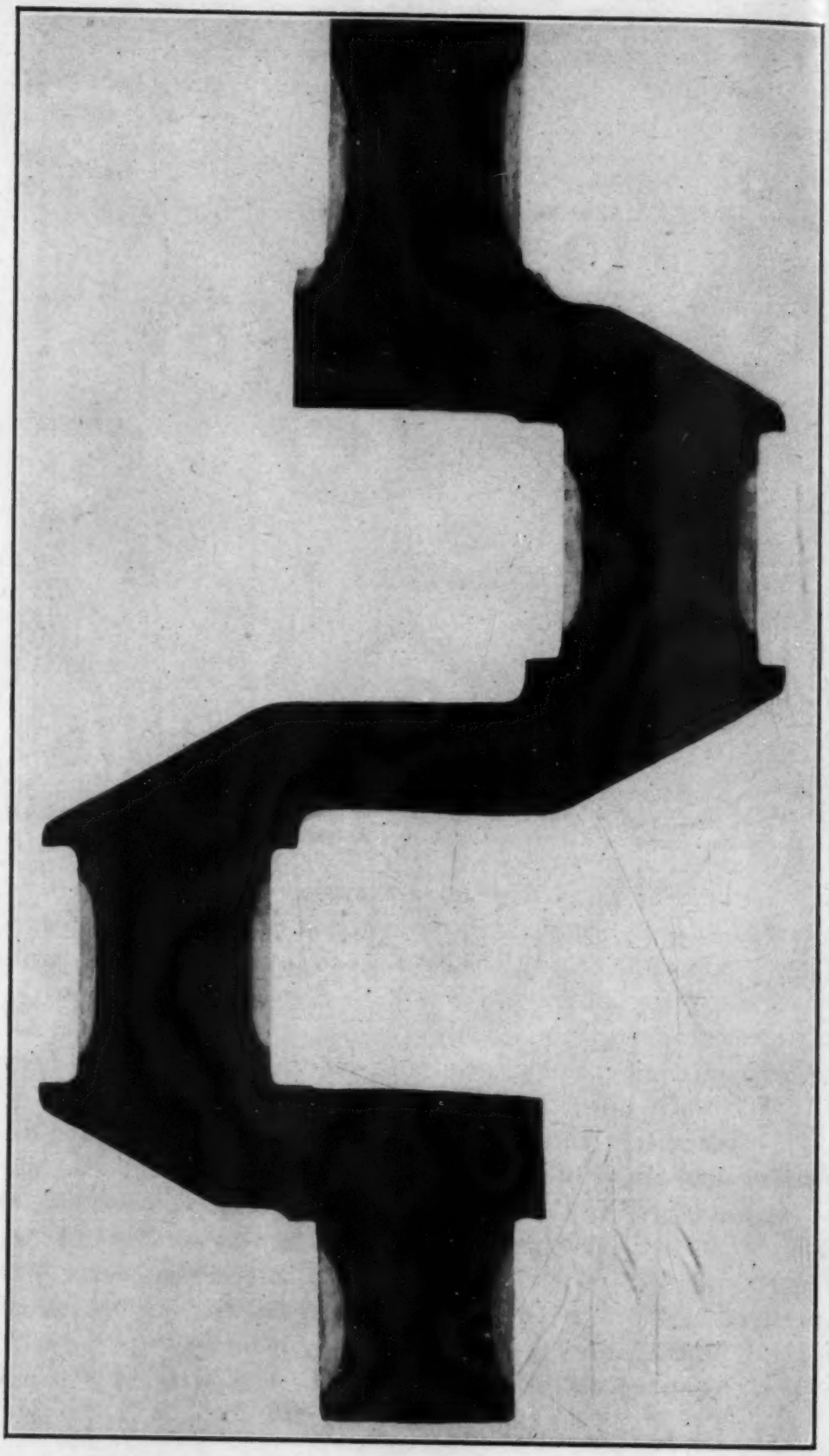

Fig. 5—Photograph of a Longitudinal Section of a Refrigerator Compressor Crankshaft in Which the Thrust Wall Has Been Hardened Leaving the Included Fillet Untouched.

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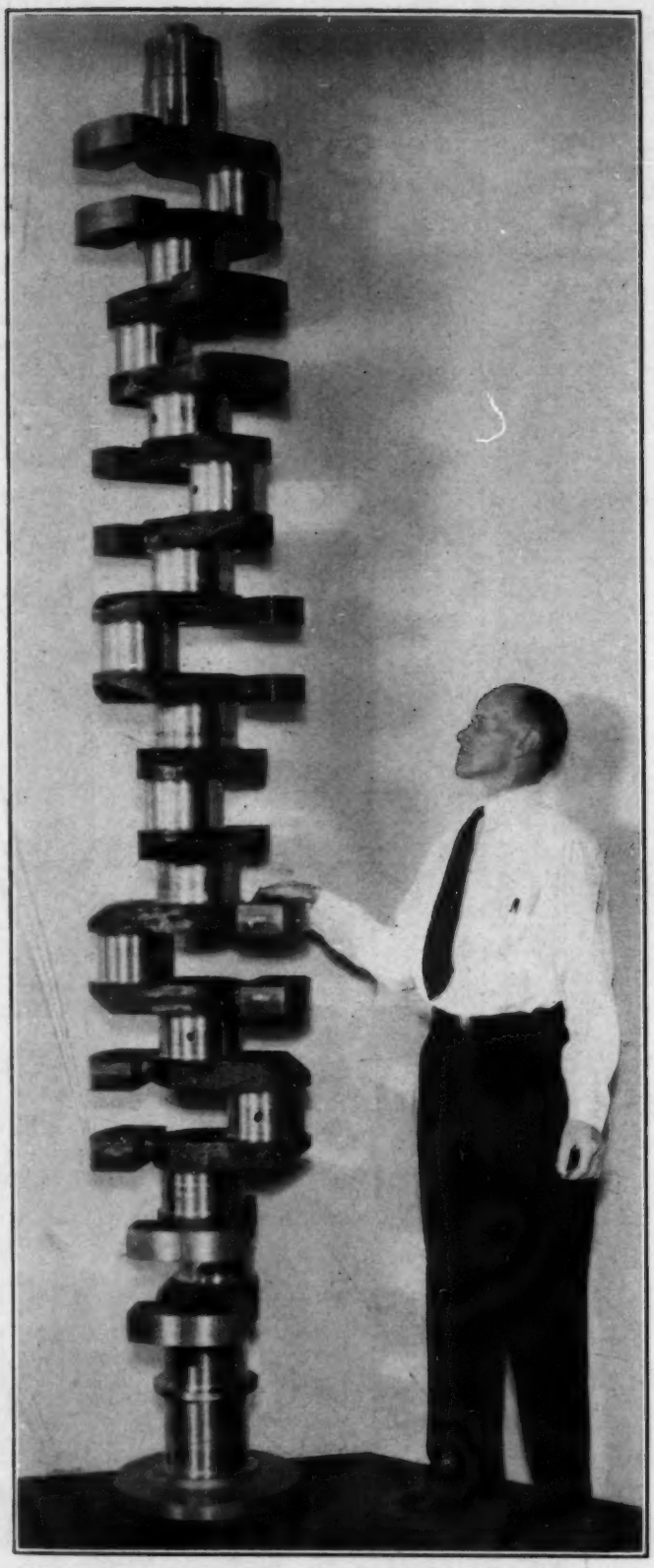

Fig. 6—Largest Die-Forged Crankshaft Ever Produced. Weight—2000 Pounds. 6.5-Inch Main Bearings, All Induction Hardened. Average Heating Time per Bearing 15 Seconds.

Table I
Chemical Analysis of Crankshaft Steels That Can Be Induction Hardened

No.	Type	Carbon	Mn	P Max.	S Max.	Nickel	Chromium	Molybdenum	Silicon Max.	Copper
1	S.A.E. No. 1045	0.40/0.50	0.60/0.90	0.040	0.050	.....	.....	.....	0.030	....
2	S.A.E. No. 1050 Modified	0.45/0.55	0.70/1.05	0.040	0.050	.....	0.25 Max.	.....	0.030	....
3	S.A.E. No. T1345	0.40/0.50	1.60/1.90	0.040	0.050	.....	.....	.....	0.030	....
4	Mola.	0.588	1.16	0.014	0.015	Nil.	0.21	0.15	.....	....
5	S.A.E. No. X4340	0.35/0.45	0.50/0.80	0.040	0.040	1.25/1.75	0.70/0.90	0.15/0.25	0.030	....
6	S.A.E. No. 4140	0.35/0.45	0.50/0.80	0.040	0.040	.....	0.80/1.10	0.15/0.25	.....	....
7	S.A.E. No. X3140	0.35/0.45	0.60/0.90	0.040	0.040	1.10/1.50	0.70/0.90	.....	.....	....
8	Foreign	0.382	0.38	0.011	0.021	2.74	0.88	.....	.....	....
9	Foreign	0.32/0.38	0.30/0.50	0.020	0.030	2.50/3.00	0.80/1.00	.....	.....	....
10	Foreign	0.294	0.25	0.008	0.008	3.91	1.07	0.51	.....	....
11	Foreign	0.350	0.63	0.008	0.009	0.65	1.06	0.95	.....	....
12	Foreign	0.516	0.59	0.018	0.037	0.96	Nil.	Nil.	.....	....
13	Foreign	0.376	0.74	0.029	0.039	0.08	0.33	Nil.	.....	....
14	Ford	1.198	0.66	0.019	0.033	0.19	0.42	Nil.	0.90	1.82
15	Malleable Iron	2.50	0.93	0.170	0.095	....	....	....	0.94	....
16	Graphitic Steel	1.50	0.40	0.015	0.020	....	....	....	0.80	....

tion developed by The Ohio Crankshaft Company has been named the TOCCO process, which is a word formed by combining the first letters of the company name.

The shafts which are to be hardened are usually forged of a modified S.A.E. 1050 steel and either heat treated or normalized for maximum ductility consistent with the physical properties desired. A large number of other steels and materials have been successfully hardened by this process. Table I. The shaft is then machined to the point of final grinding with 0.015 to 0.025 inch of stock left on the bearing diameters and 0.008 to 0.010 inch of stock left on the bearing widths. The bearing surfaces are then hardened, the shaft drawn at a low temperature, and then finish-ground. The bearing hardness obtained is in the range of 58-62 Rockwell C, which is approximately

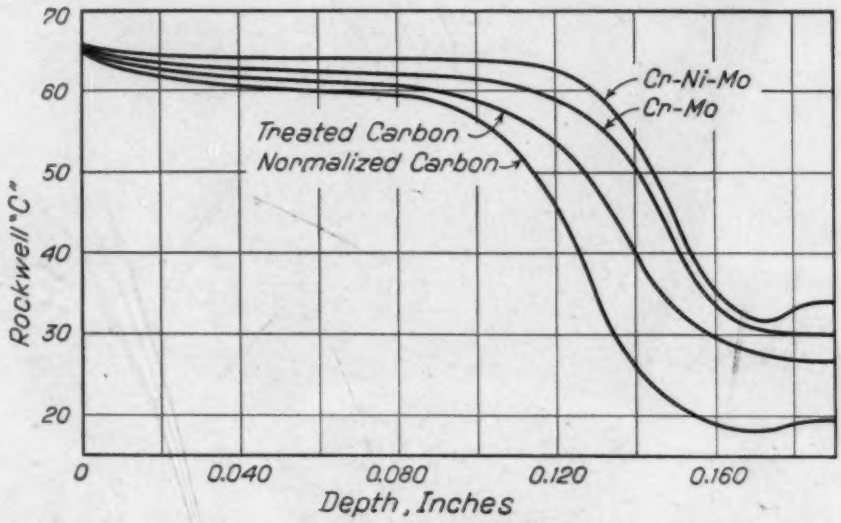

Fig. 7—Rockwell Hardness Chart Showing Retained Hardness Through 80 Per Cent of the Depth of the Hardened Area with No Drop in Hardness Below That of the Core.

80 scleroscope or 600 Brinell, and this hardness is maintained through 80 per cent of the depth of the hardened area. Fig. 7 is a Rockwell hardness chart showing retained hardness through 80 per cent of the depth of the hardened area, with no drop in hardness below that of the core.

Preference is had for steels of the McQuaid-Ehn rating of 6-8, with only a slight degree of abnormality. This slight abnormality together with the fine-grained steel, produces a martensitic structure in the hardened zone without the customary martensitic cracking.

To completely insure the success of the process, particularly in producing a good bond, the steel used should be either previously

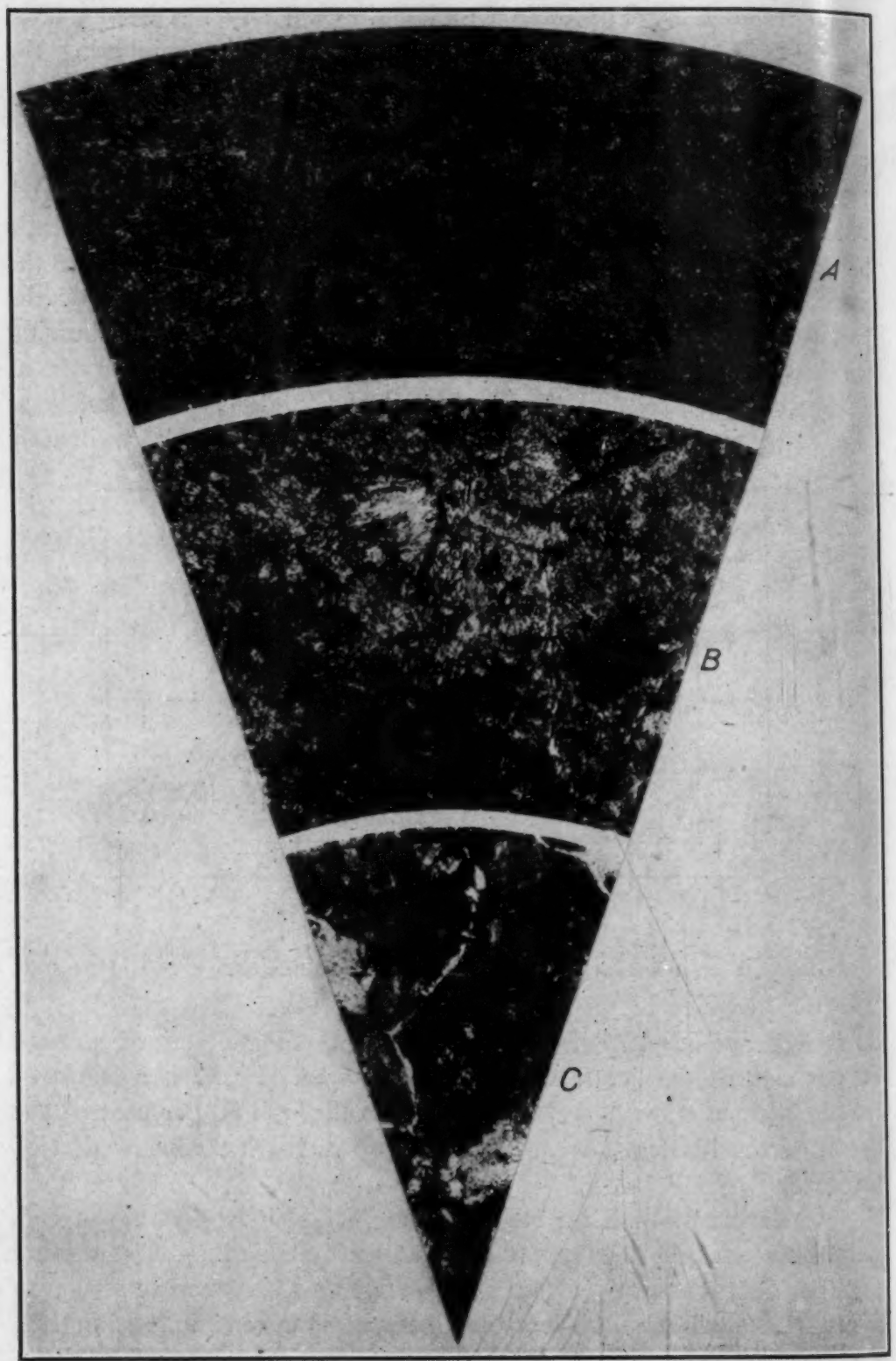

Fig. 8—S.A.E. 1050 Steel As-Forged and Induction Hardened Showing Incomplete Dispersion of Carbides Due to Too Rapid Heating with Insufficient Time for Complete Carbon Diffusion. A—Induction Hardened Zone. B—Gradation Zone. C—Unhardened Zone.

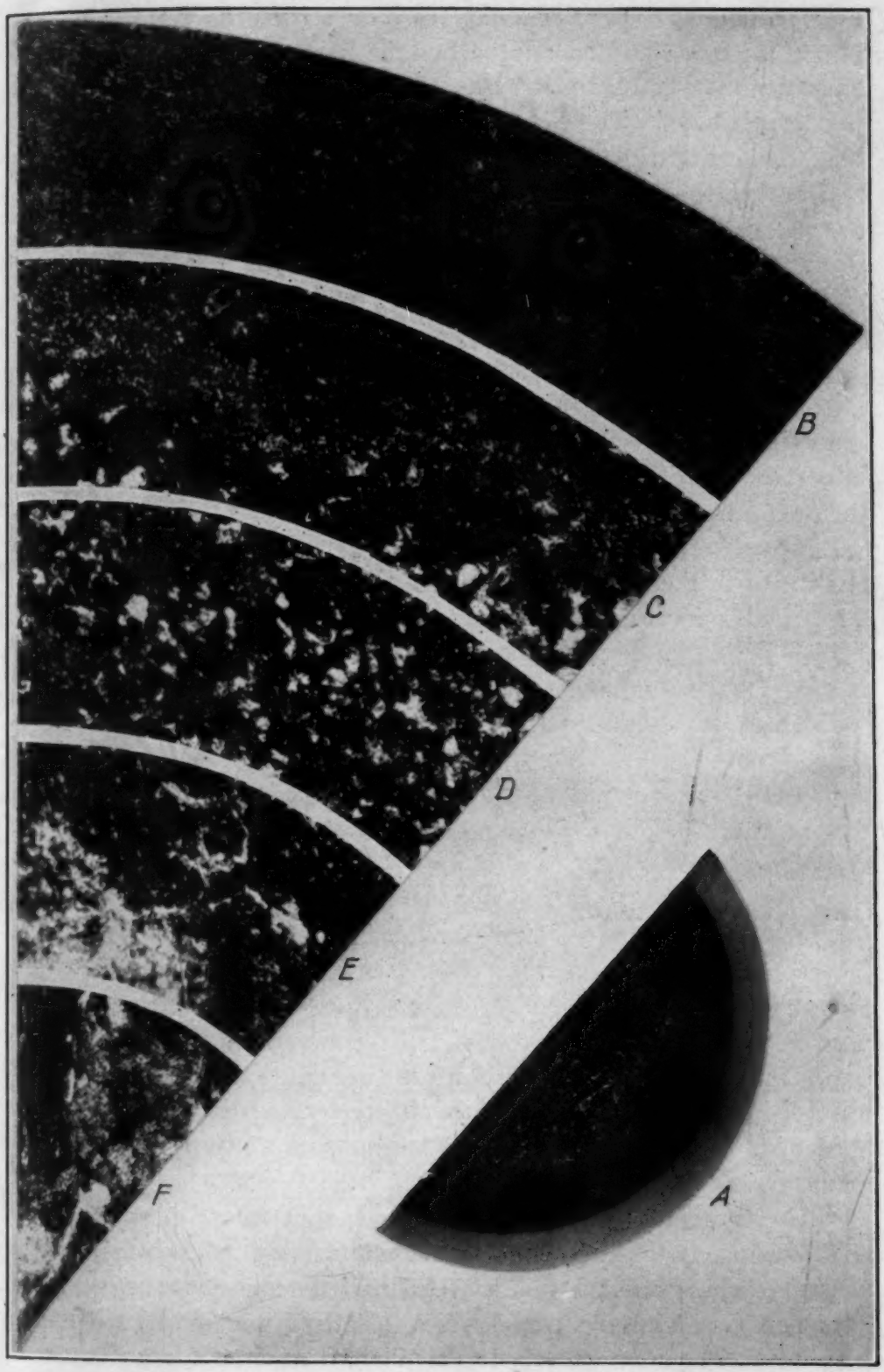

Fig. 9—Carbon Steel. 0.53 Carbon, 0.90 Manganese, 0.27 Chromium. A—Surface; B—Demarcation Zone; C—Normalized Zone; D—Demarcation Line; E—Core; F—Section. × 100.

heat treated to a predominately sorbitic structure or normalized to obtain a grain size that approaches the heat treated structure. This structure may be obtained by an accelerated cool from the normalizing temperature. The heating cycle used in this process is so rapid that it permits little or no diffusion time, which necessitates having the steel in the proper condition before hardening. Fig. 8 shows an S.A.E. 1050 as forged and induction hardened showing incomplete dispersion of carbides due to too rapid heating not allowing sufficient time for complete carbon diffusion. Another ½ second was sufficient for complete diffusion.

An interesting experiment which may open new fields is to inductively normalize an "as-forged" part and then immediately inductively harden the normalized structure. This application is illustrated in Fig. 9 in which is shown the martensitic hardened area, the fine-grained ferro-pearlitic normalized area and the coarse-grained "as-forged" structure along with the gradual blending of these structures in the demarcation zones, all on a 2-inch diameter section. The caption for Fig. 9 defines the various areas shown.

The steel used for a crankshaft which is to be subsequently induction hardened must be free from surface imperfections and the micro-constituents should show a minimum tendency toward segregating into bands. For the reasons previously described, the heat progresses inward with little rise in surface temperature above the Ac_3 point so that quenching is instantaneous from just above the critical point.

The customary surface decarburization and grain growth does not occur, due to the rapidity of the heating cycle which is measured in seconds and tenths of seconds. The resulting martensite is extremely fine-grained, even at 1000 diameters, and the usual large needles are absent, thus producing exceptional ductility throughout the hardened area. Fig. 10 shows the structure at 100 diameters of an S.A.E. 1050 normalized with an accelerated cool and induction hardened. Fig. 11 shows the same specimen at a magnification of 500 diameters.

The combination of accurate control, split-second heating cycles, and pressure-quenching applied instantaneously at the end of the heating cycle, produces a bond—that is, the cohesion between the hardened area and the core—which blends gradually into the core structure without any marked precipitation of free ferrite either in or below the gradation zone. The gradual blending of the hardened

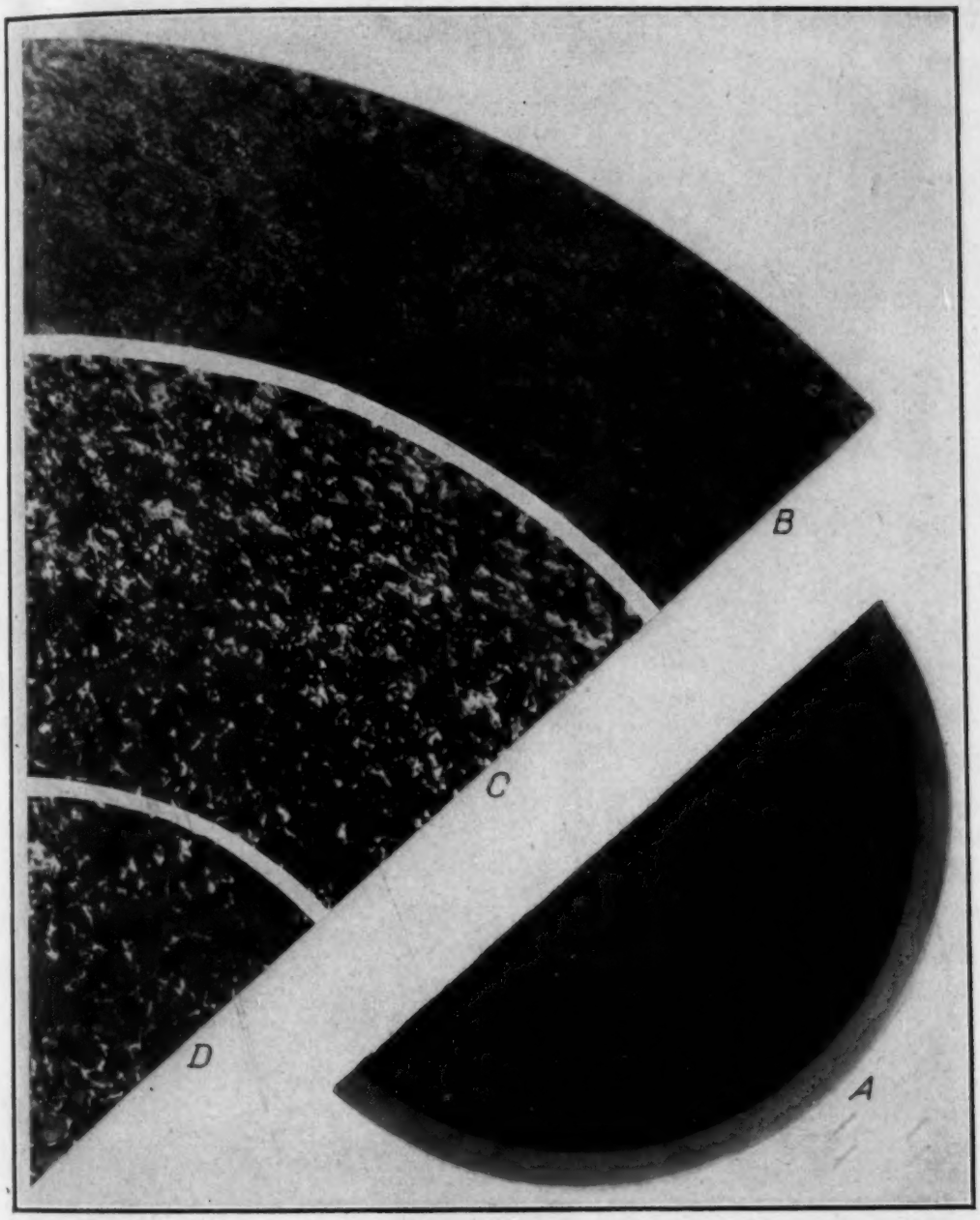

Fig. 10—S.A.E. 1050 Steel Normalized with an Accelerated Cool and Induction Hardened. A—Section; B—Surface; C—Gradation Zone; D—Core. Analysis of Steel—Carbon 0.53, Manganese 0.90, Chromium 0.27. Physical Properties of Core—Elastic Limit 50,000. Ultimate Strength 100,000. Reduction of Area 40.0. Elongation 17.0. Izod Impact 25. Brinell 207. Grain Size 6-8.

area into the core without precipitation of ferrite prevents any flaking or spalling in service. Fig. 10 shows the gradual blending of structure through the demarcation zone.

The original structures in the fillet and the core remain entirely undisturbed since the heating is confined to the areas desired hard-

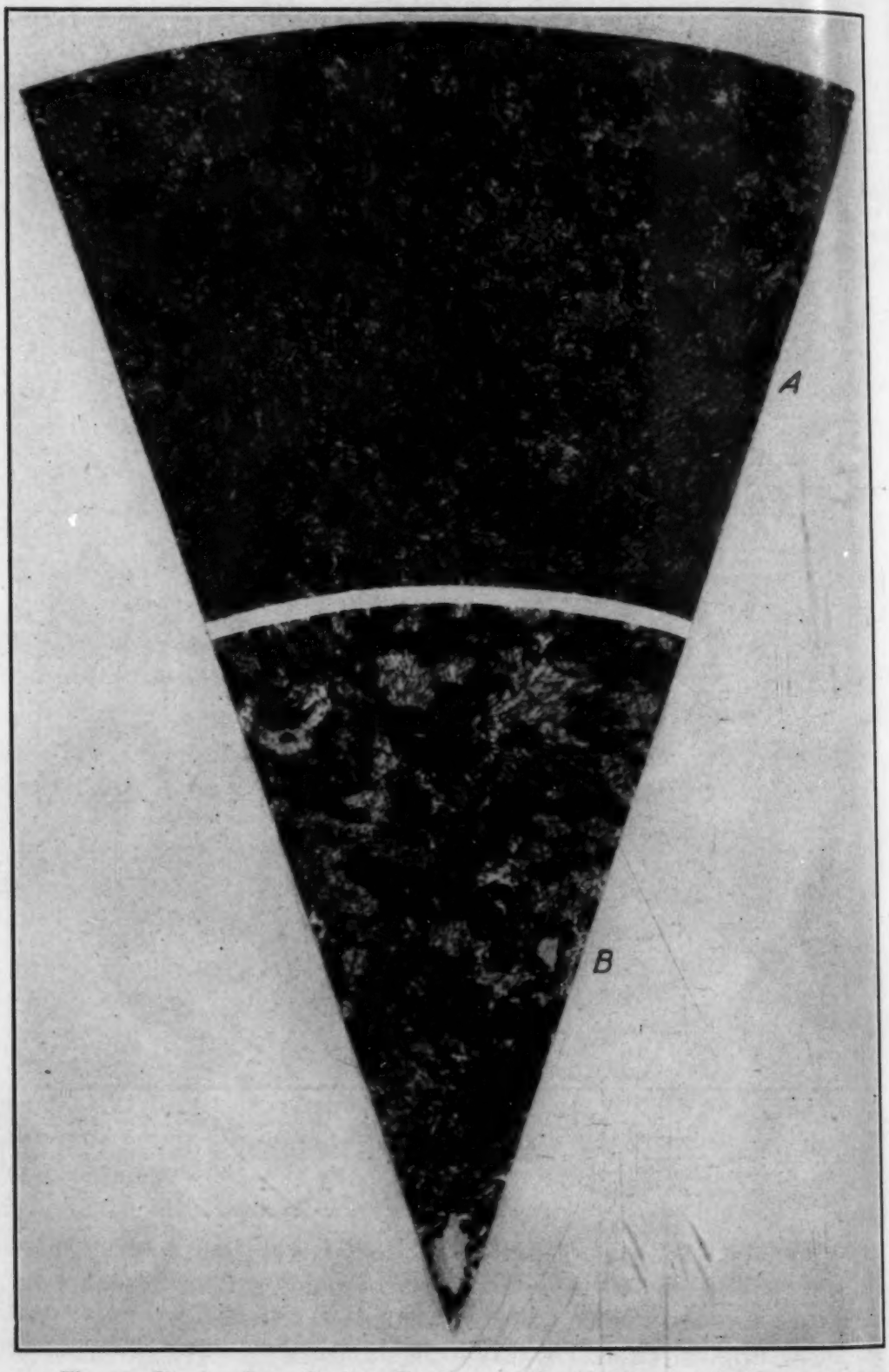

Fig. 11—Showing Same Steel as Fig. 10 at a Magnification of 500 Diameters. A—Surface; B—Demarcation Zone.

ened due to the design of the inductor block and the skin effect peculiar to high-frequency current. Since the fillets are not hardened but left in their original ductile condition, stress concentration, therefore, is decreased. Many engineers place considerable emphasis on fatigue values as determined in the fatigue testing machine without giving proper consideration to stress concentration. It is a well-known fact that alloy steels produce better fatigue values than carbon steels in the testing machine, but they also show a greater tendency to stress concentration.

Since stress concentration diminishes with increasing ductility, a crankshaft of maximum ductility combined with high bearing hardness of the proper structure at approximately 60 Rockwell C is ideal for resistance to wear and the ability to withstand physical abuse.

As an illustration of this point, an engine manufacturer using an alloy crankshaft heat treated it to the limit of machinability and found that the shafts failed after about 30,000 miles. The failures were analyzed as being caused by fatigue due to stress concentration in the fillets. Without changing any factor except the crankshaft steel, which was changed to modified S.A.E. 1050 steel, and with the bearings induction hardened for wear resistance, the failures ceased. Incidentally, there are many records of these shafts running 150,000 miles without perceptible journal wear.

We can now, therefore, approach the ideal crankshaft—one with maximum ductility to withstand physical abuse and with high bearing hardness to withstand wear.

A further development, where crankshaft design permits, is to machine the forging in the accelerated normalized condition instead of the heat treated condition, induction harden and finish-grind without further heat treatment. This method presents the obvious advantage of saving in heat treating costs. The normalized structure is easier to machine, saving in machining costs and tool life and, furthermore, it does not require as many or as costly straightening operations during machining. This development permits the use of the hard surface shaft with little or no increase in cost.

Due to the fact that new combinations of bearing materials with the surface-hardened shaft will permit higher bearing speeds and pressures, the newer engines can be designed to take full advantage of these features with a consequent increase in horsepower

per cubic inch of displacement or, stated another way, the weight per horsepower can be decreased.

Summary

The salient features of this process may be summarized as follows:

The fine grain martensitic structure is accurately controlled, both as to depth and width with no change in the adjacent structure. The area between the hardened zone and the core is well diffused. The control factors, reproduced automatically, insure absolute uniformity and duplication of the results.

Because of the development of the induction process, it is now practical, where crankshaft design permits, to use properly cooled normalized crankshafts in place of heat treated crankshafts in the automotive field. In the heavy duty field, carbon steels may be substituted for alloy steels and it is within the realm of possibility that cheaper steels will be used in aviation engine crankshafts. High bearing hardness is produced without impairing the ductility of the shaft. In applications where high quality and uniformity are essential (of which crankshafts are an excellent example), the use of this method insures high bearing hardness on a shaft designed for maximum ductility with the result that the shaft has long bearing life as well as increased resistance to stress concentrations. In brief, maximum resistance to wear can now be combined with maximum ductility to produce the ideal crankshaft.

The advantages of a split second heating cycle over customary long heat cycle methods, both from metallurgical and production standpoints, need no special comment.

There is no anticipation that this hardening method will completely replace other methods of producing high surface hardness. It is, however, a new tool for the metallurgist and the motor designer to be used on those applications where investigation merits its use.

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